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PREPARATION AND TESTING OF EXPLOSIVES

March 15, 1944 to April 15, 1944

Copy No.	
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Report Compiled by
H. J. Fisher

This is a compilation of informal reports pertinent to Projects NO-199, NO-201, NO-211, NO-231, NO-232, OD-01, OD-04, OD-103, OD-109, and OD-118 submitted in advance of formal reports. It is not to be presumed that the work is complete or that the results herein reported are other than tentative.

Ralph Connor
Chief - Division 8

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Note:

1. Work on the subject "Controlled Decomposition of Nitrocellulose" is reported in the Division 8 Interim Report on "Special Propellants". Formal Progress Reports on the above subject have been prepared at Columbia and Ohio State University and will be issued soon.

2. Work on the general subject "Nitramines" is reported in Division 8 Interim Report on "Organic Development Problems".

EXPLOSIVE COMPOSITIONS

Report Submitted by
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Abstract

1. A 5000# batch of PEP-2 has been manufactured at the duPont Eastern Laboratory. The outstanding difficulty revealed in this production was the slowness of rolling of this material, compared with rolling dry Composition C or Composition C-2.

After 3 months' storage at 60°C., the superiority of British Arachis-nut lecithin over American soy-bean lecithin for RDX Polar P.E. continues to be demonstrated.

2. Exudation tests and bullet tests with caliber 0.50 ball, M2 show that substitution of paraffin for Aristowax-alox in Composition B will neither increase the exudation nor the bullet sensitivity.

The effects of diluting Composition B from a composition 60/40/1 RDX-TNT-Wax to 55/45/1 RDX-TNT-Wax upon the viscosity and the power have been determined.

Experiments on the segregation of RDX and of wax in large castings of Composition B have been begun.

3. The relative insensitivity of the liquid explosive Methylite-20[1] to repeated light blows has been demonstrated by shaking a sample for 8 hours in a vigorous shaking machine, without explosion.

4. It has been decided that the acetone extract of lecithin is the most satisfactory surface-active agent which has been found for Ednatols. Its effects on the viscosity of Ednatol slurries has been measured with an efflux viscometer under various conditions, and its effect on the vacuum stability has been investigated This extract also decreases the viscosity of DBX and Baronal slurries, but when added to Gulf Crown Oil E does not make a good plastic explosive with RDX, as does both the whole lecithin and the lecithin residue.

5. Three different lining and padding combinations, which have been applied to cases of the Aerial Depth Bomb, AN-Mk47, in an attempt to desensitize it to hard-surface impact, are described.

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[1] 80% Nitroglycerine/20% dimethyl phthalate stabilized with 0.5% ethyl Centralite.

Report

1. Plastic Explosives

(1) PEP-2

A 500# batch of PEP-2 with 14% oil has been manufactured at the duPont Eastern Laboratory. In this preparation the wet PETN was added to the Crown Oil E in the incorporator with stirring at room temperature. Enough PETN was added to give a final composition of 86% PETN + 14% oil. Before rolling the wet product has 10-15% water, since this is the water content of the wet PETN. The rolls were set at a roll separation of 0.004" with spring pressure of 2000# at one end and 1400# at the other. The wet oiled mixture was passed 10 times through the rolls. However, through some misunderstanding, the first 200 lbs. of wet PEP-2 were passed 20 times through the rolls. This 20 pass material was put in with the regular 10 pass material for the first 650# drying batch. The final dried product was a good, firm plastic, but it did seem, immediately after drying, to be somewhat shorter than the previous experimental sample rolled the same way. On standing about 2 days, however, the product softened somewhat and its length increased. It will pass extensibility specifications even after it is removed from the drier.

Analysis of the first two oiled batches showed 14.4% and 13.8% oil respectively. It is Dr. Burrows' belief that in large scale production, it would be difficult to hold the oil concentration to much better than 14% ± 1%. The water content before rolling was about 15%, and after rolling, 3.5% and 4.2% for the two oiled batches.

The principal difficulty experienced in manufacturing this plastic occurs in rolling. The wet, oiled PETN pressed through the rolls very easily the first 2 or 3 times, but after that it becomes firm and must be forced through the rolls. The water which is squeezed out of the material lubricates the surface of the rolls so that they cannot grip the material and pull it through.

In an effort to remove some of the water which is trapped in the oiled PETN during oiling, the material was oiled hot. It was hoped that hot oiling would bring the water to the surface, where it could be decanted. This method did not help very much, possibly because the material was not allowed to get hot enough.

The only other variation in procedure which was possible with the equipment at hand was to oil some PETN from a slurry. For this purpose, a small laboratory incorporator was used to slurry-oil 8 lbs. of PETN. This material was cooled, filtered and rolled without any visible improvement.

It will probably not be advantageous to heat the rolls and roll PEP-2 hot, because PEP-2 does not soften up on heating.

The maximum possible production of PEP-2 per shift (about 6-1/2 hours of actual rolling) on one roll with the present procedure is 500 lbs. This is about the rate of production of wet Composition C which can be turned out at the rate of 80-100 lbs. an hour. When Composition C is rolled dry, one roll can turn out 250 lbs. an hour. Composition C-2, however, can be turned out at the rate of 1000 lbs. an hour by one roll.

Dr. Burrows did not think it would be advisable to roll dry PEP-2 because of its sensitivity.

Some work will be done, soon, at Bruceton to determine the effects of water and method of incorporation on rolling on our roll mill.

(2) Storage Tests of RDX Polar P.E. Made with British Lecithin

After 3 months' storage at 60°C. in closed and open containers, the samples of RDX Polar P.E. made with British lecithin still show no signs of deterioration in plastic properties. The samples are somewhat firmer than when first made, but are perhaps of better consistency because of this fact, since they were rather soft when first made. As has been mentioned previously (PT-18, page 5 and PT-19, page 4) samples made with American soy-bean lecithin deteriorated rapidly at 60°C.

2. Composition B

(1) The Use of Paraffin in Composition B

(a) Exudation Tests (L. Goodman)

Exudation tests were made with 1" cylinders, 1/2" high of cast Composition B, which were held at 60°C. on a stack of filter-papers in a weighing bottle. The loss in weight of the pellets after one week has been determined, to compare the behavior of Composition B with paraffin and that of regular Composition B made with 90/10 Aristowax-Alox. The following results were obtained:

Pellet	% Loss in Weight
Composition B (Aristowax-Alox)	
Sample I	0.281
Sample II	0.415
Composition B (Paraffin)	
Sample I	0.110
Sample II	0.113

These values are all somewhat high, as the result of a certain amount of loss of TNT by sublimation, but it seems clear that the substitution of paraffin for Aristowax-Alox in Composition B should not increase exudation.

(b) Bullet Sensitivity (W. H. Rogers)

The relative sensitivities to bullet impact of Composition B made with Aristowax-Alox and with paraffin have been further studied by trials in which the materials were tested, cast in 1-1/2" pipe-nipples, using the caliber 0.50 ball, M-2 bullet. The following results were obtained:

Explosive	No.	N.E.	E.P.	E.	E.C.
Composition B(Aristowax-Alox)	25	9	12	4	-
Composition B(Paraffin)	30	18	8	1	3

Under these conditions it appears that the material made with paraffin is, if anything, somewhat less sensitive than the regular Composition B, whereas the more extensive tests made with the caliber 0.30 ball, M-2 bullet indicated the two to be of about equal sensitivity. In any case, it seems clear that a change from Aristowax-Alox to paraffin would have no harmful effect on bullet sensitivity.

(2) Properties of 55/45 Composition B

In connection with a proposal to lower the RDX concentration in Composition B from around 60% to around 55% we have measured the drop in viscosity and the decrease in power (Ballistic Mortar) attending such a change.

The decrease in viscosity will depend on the initial viscosity of the 60% slurry, being the greater, the higher is this value. The following table gives the ratio of the viscosities at 100°C of 60% and 55% RDX slurries (otherwise identical) for a few different values of the viscosity of the 60% slurry[1].

η (60%) 100°C	η (60%) / η (55%) 100°
5 poises	3.0
10 "	4.0
25 "	5.6
50 "	8.3

These values were checked by diluting a slurry of initial viscosity 23.2 poises from 60% RDX to 55%. The viscosity was reduced to 4.8 poises, giving a ratio of 4.9.

The decrease in powder, as measured by the Ballistic Mortar, attending dilution of a Cyclotol from 60/40 to 55/45 will be expected to be about 2 T.V. units, or 1.5%, as determined by measurements of the power of a series of Cyclotols of varying compositions.

(3) Segregation of RDX in Composition B

It has been pointed out to us by Lt. Comdr. Stephen Brunauer that the British have made experiments on sectioning large castings of Composition B and Torpex, and find areas from which TNT has drawn away on cooling, leaving abnormally high concentrations of RDX. They regard the role of wax as a phlegmatizer in such mixtures as that of a desensitizer of these areas. We have made 2 cylindrical castings of Composition B's of 8" diameter and about 10" high, and inspected them for high RDX areas. In one of these castings, which analyzed 53.3% RDX in bulk, there was a central section markedly lighter in color and porous in appearance, which contained 66.1% RDX. The other casting showed a similar segregation, but was not analyzed. A similar casting was made with a 60/40 Cyclotol; this casting cavitated instead of segregating at the core, apparently because the material happened to be less viscous, and perhaps because the conditions of cooling were somewhat different. It is planned to prepare more castings, with and without wax, of

[1] This table has been constructed from general results on the concentration effect on the viscosities of Cyclotols, and involves many measurements of a large number of slurries.

similar viscosities and cooled under identical conditions to investigate this matter under controlled conditions.

3. Liquid Explosives: Sensitivity of Methylite 20 to Shaking

Various tests of the sensitivity of Methylite-20 to such severe blows as those provided by bullet-impact, dropping of containers, and striking containers with a heavy pendulum have been made. It was decided to arrange a shaking test to determine its sensitivity to many repeated blows of mild nature. A pint paint can, half filled with Methylite 20, with 16 ball-mill stones added, was covered and attached to the platform of a shaking machine. The can was subjected to 8 hours of shaking through a 6" amplitude, at the rate of 172 shakes per minute, without detonation.

4. The Effect of Surface-Active Agents on the Pourability of Ednatols (O. Bolduan)

(1) Quaternary Ammonium Salts

Two quaternary ammonium salts, triethyl cetyl ammonium bromide and dimethyl ethyl cetyl ammonium bromide were received from Onyx Oil and Chemical Company. These were claimed to be chemically pure. Both of these compounds improved the pourability, but the dimethyl ethyl cetyl ammonium bromide reacted immediately with TNT to give a red color, and the trimethyl cetyl ammonium bromide reacted slowly upon standing with molten TNT. The pH of a saturated aqueous solution of the dimethyl ethyl compound was 7.6, indicating the presence of free amine, while the trimethyl compound had a pH of 3.9. Vacuum Stability tests run on a slurry containing 0.3% of the trimethyl compound, indicated excessive gas evolution.

(2) Other Surface Active Agents

A series of cationically active surface-active agents, derived from triethylene tetramine condensed with fatty acids, supplied by Synthetic Chemicals Company, were investigated. All of them reacted with TNT.

A glyceride supplied by Hachmeister, Inc., was tried. No obvious effect on the pourability was observed.

(3) Lecithin Extract

To date, the best surface active agent for Ednatols appears to be the acetone extract of lecithin. Consequently, it was decided to evaluate this agent in a more quantitative manner.

(a) Viscometry

The modified Stormer viscometer is not well suited for measurements of viscosities of flocculated slurries because they have a tendency to segregate around the stirrer, but when the agent has been added, then the stirring tends to whip air into the slurry, since the foam is stabilized by the agent. Accordingly, an efflux viscometer previously built for Cyclotol studies was used in this investigation. The diameter of the tube is 6 cm., the diameter of the orifice 1 cm., and the length of the orifice tube 4.7 cm. The tube is placed in a water bath which is heated electrically, maintaining a temperature of 100°C. The standard procedure adopted was to place a rubber plug, connected to a stiff wire, into the orifice; add 200 grams of slurry into the viscometer; allow the slurry to attain a temperature between 99°-100°; then pull the plug and observed the time for 50 grams to flow into a beaker on a balance below the orifice.

A 57% Ednatol slurry, using standard duPont pilot-plant Haleite[2], was placed in the tube. When plug was pulled, only the slurry directly over the orifice dropped through while the rest of the Haleite on the sides formed a loose network through which only TNT drained. Flow stopped before 50 grams had been collected. Another 57% slurry was made up with the addition of 0.36% acetone extract of lecithin. The efflux time for 50 grams was 4.9 seconds. The qualitative appearance of the flow was entirely different, there being no tendency to set into a semi-rigid "network," as in the absence of agent.

Now the effect of this "network" of crystals will be most important when flow occurs under small shearing stresses, as in this small efflux viscometer; in pouring from large kettles, where the shearing stresses are high, the effects of flocculation will probably be smaller. Accordingly, efflux experiments were also done in which the slurry was stirred vigorously with a motor-driven ring-stirrer, which tends to break up the Haleite network mechanically, in the hope of obtaining results which would be more representative of the comparative behaviors of treated and untreated Ednatols in large batches. The following table gives results, with and without stirring, for several Ednatols without added agent, and for a 57% Ednatol with the lecithin-extract as agent, in the amount of 0.3%.

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[2]This material has the following grist:

On #30	1.8%
#30-50	16.3%
#50-100	81.2%
#100-200	1.7%

Time of Efflux of 50 Grams from 200 Grams in the Efflux Viscometer

Composition	No Stirring	Stirring
50% Haleite no agent	2.7 sec.	4.4 sec.
52% Haleite " "	3.5 sec.	4.4 sec.
52% " " "	-	4.8 sec.
57% " " "	(50g will not flow out)	14.6 sec.
57% " agent added	4.9 sec.	6.5 sec.

These results are not sufficiently extensive to be conclusive, but they suggest that a 57% Ednatol with about 0.3% of this agent will have an efflux rate with stirring in this test, about the same as that of a 53-54% slurry without agent. Compared without stirring, the effect of the additive will be made more striking, but it is not clear just which test is representative of the differences which will be found in large-scale pouring. Further tests, including pouring-tests from kettles, will be made.

(b) Stability

Samples of Ednatols containing 0.3% acetone-extract of lecithin have been compared with untreated material, in the 100°C Vacuum Stability test, with the following results:

100°C. Vacuum Stability	Gas evolved in 48 hr. (cc.)
Ednatol (untreated)	0.76
Ednatol + 0.3% lecithin extract	1.68
" " " "	2.02

The addition appears to increase the gas volume but it is not made as large as that from, say, 50/50 Pentolite.

(c) The Effects of Lecithin Extract on Other Materials

It has been found that the lecithin extract improves the pourability of DBX and Baronal; the effect was noted when the agent was added to mixtures prepared with very finely-divided ammonium and barium nitrates.

Samples of dry-milled RDX Polar PE have been made using oils which were 95/5 Gulf Crown Oil E - Acetone Extract of Lecithin and 95/5 Gulf Crown Oil E - Acetone Residue of Lecithin, in an attempt to see whether in this application of lecithin (the normal oil is 95/5 Gulf Crown Oil E - Lecithin) the acetone

extract might also be the active ingredient. The converse appears to be the case, for only the acetone residue gave a good plastic.

5. Desensitization of Service Munitions

The purpose of this project, which has been given the title "Desensitization of Service Munitions" is to develop methods of diminishing the drop and bullet sensitivity of depth bombs, G.P. bombs, and other service munitions, particularly of munitions loaded with aluminized explosives. The following constitutes a brief report of the preparation of lined and nose-padded cases of the A. D. Bomb, AN-Mk47, which are to be loaded with Torpex II at the Naval Mine Depot, Yorktown, Virginia, and subjected to the hard-surface drop test at the Aberdeen Proving Ground. Three types of linings, ten bombs each, were prepared.

(1) ERL Experimental Lining, Type A

These bombs have been given a nose pad, about 1-1/2" thick at the center, and a 1/4" thick wall lining of a soft and rubbery asphalt, called "H.H.Robertson Compound No. SR1741." This material is a bituminous-like compound, characterized by having a high viscosity index, and being of a rubbery nature over a wide temperature range. The following are of particular interest among the data comprising its specifications:

(a) Softening Point ASTM D-36 245 to 260°F.

(b) Penetration ASTM D-5

(1) at 77°F. 30 to 38
(2) at 32°F. 23 to 30

(c) Recommended Pouring Temp. 450°F.

This material, when mixed with Torpex II does not increase its gas evolution in the Vacuum Stability Test at 120°C., and it is felt that its softening point is high enough so the linings will not flow during loading of Torpex II.

The wall-lining was applied by heating the bomb case to 120°F. in an oven, transferring it to a rotating device with its cylinder-axis horizontal, adding 10# SR-1741 at 460°F., and rotating rapidly for from 10 to 15 minutes, after which the lining was cool enough not to flow further. The nose pad was formed by standing the bomb on its nose with the cylinder axis vertical and pouring in 5# of SR-1741 at 460°F., using a special funnel to by-pass the hydrostatic fuze and booster tube. The

remaining length of the nose-fuze well was covered by a boot of 1/4" cork (Armstrong 9114) covered on the outside with SR-1741.

The lining will decrease the cavity volume by about 10%, i.e., to 3712 cu.in.

(2) ERL Experimental Lining Type B (Ten bombs)

This lining is identical with Type A, except that it has a 10# nose pad, about 2-1/2" thick at the center.

This lining will decrease the cavity volume by about 13%, i.e., to 3574 cu.in.

(3) ERL Experimental Lining Type C (Ten bombs)

These bombs have been given a nose pad, about 2" thick at the center, and a 1/8" thick wall lining, of a low density, fine grain, cork sheet called Armstrong 9114. This is a material of quite different nature than any which has been already tried, and is known for its excellent shock-absorbing properties.

The nose-pad was built up from eight discs of 1/4" sheet of appropriate diameters, glued together with 3M Elastic Cement EC-260 (Minnesota Mining and Manufacturing Company). The wall-lining of 1/8" sheet was made in two pieces, and glued to the walls at the joints with the same cement. The entire cavity was finally painted with Black Anti-acid Cavity Paint, Navy Spec. OS-1356 (Pruett-Schaffer), special attention being given to all joints. The nose-fuze wall was covered with a 1/4" cork boot, also painted with cavity paint.

This lining will decrease the cavity volume by about 8.5%, i.e., to 3772 cu.in.

PHYSICAL TESTING

Report Submitted by

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1. Impact and Mortar (R. Davis)

During the five-week period between March 13 and April 15, 1944, 9216 shots were fired on 130 samples in the various impact machines. Most of these samples were of purely local interest or are reported elsewhere.

Work has been almost completed on a series of samples of twelve common explosives which have been sent out by the Canadian National Research Council to a large group of laboratories which measure impact sensitivity. At ERL their sensitivities have been measured on Impact Machine Designs No. 3, 12, 126, and 13. The collected results should be available next month.

Much time has been spent during the past month in assembling a special report on impact sensitivity work at the ERL, which will give detailed descriptions of the various machine designs and results obtained with each.

2. Bullet Sensitivity Tests (W. H. Rogers)

(1) Studies of the Effect of the Container on Bullet Sensitivity

(a) Effect of Diameter of Container

Last month, tests were reported which permitted comparison of the sensitivities of explosives cast in containers of varying diameter and wall thickness but constant height. It was hoped that this month a report of tests made at the upper limit of this scale could be given. Pentolite was cast in five-inch nipples three inches high and caps were screwed on. Three such charges were tested and, although the first two clearly passed, the third detonated with enough violence to damage the bomb-proof seriously. Examination of the inert cases revealed that the bullet in both instances had penetrated about three and a half inches, powdering the explosive for at least a third of the total volume. It has been judged unwise to make further tests of such large charges until a stronger testing chamber is available. The results of only three shots are of little significance in themselves.

(b) Effect of Scale of Container

The effect of doubling the linear dimensions of a container was studied by comparing the sensitivity to impact of caliber 0.30, ball, M-2, bullets of 50/50 Pentolite cast in 1" pipe-nipples, 3" long, with that of the same material cast in 2" pipe-nipples, 6" long. The wall-thickness, of course, was not increased by a factor of two by this method. The results were the following:

Pentolite, 50/50 Cast	No.	N.E.	E.P.	E.	E.C.
1" p.n., 3" long	40	30 (75%)	3 (7.5%)	2 (5%)	5 (12.5%)
2" p.n., 6" long	20	7 (35%)	11 (55%)	1 (5%)	1 (5%)

The partials in this 2" series are not so violent, on the whole, as those obtained with the 1" containers; there was much evidence of burning, instead of the usual quick flash.

(c) Future Work

It is felt that these results make it clear that rather small changes in the shape, size, and wall-thickness of the container may cause significant changes in the sensitivities of the loaded containers. On the other hand, there seems to be no regularity or apparent simple interpretation of the results which have been obtained. Accordingly it has been decided to abandon the use of pipe-nipples for these tests, and to prepare special containers whose wall-thicknesses may also be systematically varied, or held constant for a variety of container shapes and sizes.

(2) Bullet Sensitivity of Aluminized Explosives

As a part of an extensive program for study of Aluminized Explosives at ERL, the bullet sensitivities of Torpex II (unwaxed), Minol II, DBX (unwaxed), and UWE (80/20 TNT - Al) have been studied, using Composition B as a comparison explosive of the non-aluminized type. These Aluminized Explosives had not been tested extensively by this method at ERL, so it was decided to disregard all old data (such as that reported in OSRD Report No. 3156) and begin anew. These explosives have been tested cast in 2" pipe-nipples, 3" long, and cast in 3 x 3 x 3" boxes of 20 gauge steel. A few tests using 3 x 3 x 3" boxes provided with heavy steel back-plates showed that steel backing does not alter the results.

Careful observation of the "partial explosions" obtained with these materials revealed that it is necessary to distinguish two types of partial explosions. One type, which will be called Type I, is that typical of Aluminized Explosives, characterized by a large, bright flare and absence of noise or serious damage to the container. The other, which will be called Type II, is the normal partial explosion, as found with all ordinary explosives. The Type II partial is generally more violent, and more of a real explosion. The results of bullet tests, to date, are the following:

Cal. 0.30, ball, M-2; 2" pipe-nipples, 3" long

Explosive	No.	N.E.	E.P.$_I$	E.P.$_{II}$	E.	E-C.
Composition B	20	19 (95%)	-	1 (5%)	-	-
U.W.E.	20	12 (60%)	4 (20%)	4 (20%)	-	-
Minol II	20	-	10 (50%)	10 (50%)	-	-
DBX (unwaxed)	20	-	6 (30%)	14 (70%)	-	-
Torpex II (unwaxed)	19	-	6 (31.6%)	12 (63.2%)	-	1 (5.2%)

Cal. 0.30, ball, M-2; 3 x 3 x 3" boxes

Composition B	20	20 (100%)	-	-	-	-
U.W.E.	20	19 (95%)	1 (5%)	-	-	-
Minol II	21	15 (71.4%)	5 (23.8%)	1 (4.8%)	-	-
DBX (unwaxed)	20	14 (70%)	6 (30%)	-	-	-
Torpex II (unwaxed)	20	4 (20%)	16 (80%)	-	-	-

The pipe-nipple test seems especially suited to distinguishing Torpex II, Minol II, and DBX, as a group, from U.W.E. These three explosives are shown to be more sensitive than U.W.E. at the 95% confidence level, while no differentiation can be made among the three at this level with the number of test-shots made [1]. On the other hand, U.W.E. itself is shown to be more sensitive than Composition B at the same confidence level. To distinguish among the sensitivities of Torpex II, Minol II, and DBX in this test, either more shots would have to be fired, or the confidence level to be regarded as significant would have to be lowered. At 90% confidence level the three still are indistinguishable, while at 80%, Minol II is distinguished as the most insensitive of the three, while DBX and Torpex II are still indistinguishable.

The 3 x 3 x 3" box test is best suited to distinguishing Torpex II as the most sensitive of the explosives under test. Even at the 99% confidence level it is so distinguished. At 95% confidence level, Composition B and U.W.E. are distinguished as less sensitive than Minol II and DBX. Even at Confidence level 80%, however, these data do not distinguish between Composition B and U.W.E., or between Minol II and DBX.

Finally, combining the results of the two tests, the following order of <u>sensitivity</u> to bullet impact is found:

Torpex II > DBX > Minol II > U.W.E. > Composition B.

Each of the above distinctions is made at a confidence level of at least 95% in at least one of the container types, except that the distinction between DBX and Minol II may be made only at confidence level 80%. These two types of test will be used as the primary method for studying the phlegmatizing effects of waxes and other materials on aluminized explosives.

(3) <u>Sensitivity of Plastic Explosives to Incendiary Bullets</u>

Both Tetryl IPE and Composition C-2 (M4) were tested in 3 x 3 x 3" boxes with Incendiary bullets, the former only with caliber 0.30 Incendiary, M1, the latter with both caliber 0.30 and caliber 0.50 Incendiary, M1. In all series the appropriate front plates were attached. The results are as follows:

- -

(1) For a discussion of the use of confidence limits, and the distinction among explosives at various confidence levels, see OSRD Report No. 3156, page 36 ff.

	No.	N.E.	E.P.	E.	E-C.
Cal. 0.30 Incendiary, M1.					
Tetryl IPE - 3x3x3" boxes, 3/16" front plate	8	8	-	-	-
Comp. C-2 (M4) - 3x3x3" boxes 3/16" front plate	10	10	-	-	-
Cal. 0.50 Incendiary, M1.					
Comp. C-2 (M4) - 3x3x3" boxes 1/4" front plate	10	10	-	-	-

Apparently the conditions of this test are not severe enough to make it of very much interest. Tetryl IPE will be tested with the caliber 0.50 Incendiary bullet, however.

3. Flame Sensitivity of Explosives (C. A. Weltman)

The sensitivity to flame of several cast and pressed pure explosives and explosive mixtures has been determined. The results are summarized in the following tables:

A) Pure Explosives

Explosive	Max. Time = 10 NF	Min. Time = 10F	Av. Time	I.I.
Cast TNT	.697	~2	~1	~100
Pressed TNT	.697	~2	~1	~100
Pressed Haleite (+2% wax)	.643	.806	.725	138
Pressed Tetryl	.384	.434	.409	244
Pressed RDX (+2% wax)	.286	.434	.360	278
Pressed PETN (+2% wax)	will not continue to burn			
Cast DINA	"	"	"	"
Cast Fivonite	"	"	"	"

B) Explosive Mixtures

Explosive	Max. Time = 10 NF	Min. Time = 10F	Av. Time	I.I.
Cast Comp. B	.485	.643	.564	177
Pressed Comp. A	.384	.643	.514	195
Cast Torpex (no wax)	.434	.589	.511	196
Cast 50/50 Pentolite	Very difficult to ignite - will not continue to burn.			
Pressed 50/50 Pentolite	" "	" "	" "	
Cast Ednatol	" "	" "	" "	
Cast 75/25 Tetratol	" "	" "	" "	

TNT unfortunately comes just at the limit of times which can be measured by controlling the opening in the rotating wheel of the flame-test machine, so that the minimum time for 10 successive fires must be measured with the stopwatch, and is not well determined. It is believed that cast and pressed TNT behave in the same manner to flames.

Haleite, RDX, and PETN were all given a 2% wax coat so they could be pressed to good pellets. This probably lowers the I.I.'s of all of them, but should not change their relative sensitivities to flame (Compare RDX + 2% wax with RDX + 9% wax in Table B). It is interesting to note that the three explosives which could not be caused to continue burning were all ester-nitrates.

Among the mixtures, cast Composition B is found more inflammable than TNT, and less inflammable than RDX. The addition of Aluminum, to form Torpex, increases the flame-sensitivity. When PETN is added to TNT the inflammability is reduced until burning will not persist. The insensitivity of Ednatol and Tetratol, however, is not easy to understand, since it would appear that these mixtures are less sensitive to flame than either of their components. It is planned to try to modify the flame test so that the heated surface will be horizontal, and so hold the melting material, instead of allowing it to drip off. This may make it possible to burn some of the less inflammable mixtures.

THE EFFECT OF HEAT ON CONFINED EXPLOSIVES

Report Submitted by
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Abstract

The behavior of Composition A on heating in a closed bomb is similar to that of pure RDX.

The Tetryl booster in the Mark46 detonating fuze, used in the Mark 35-3 five inch projectile, will detonate at a temperature at least as low as 135°C. The detonation is initiated by the sensitive fulminate primer, and occurs even though the fuze is in the safe, or unarmed condition.

Report

Investigation Group: E. C. Broge and H. A. Strecker

Further experiments have been made in connection with the problem of the "cooking-off" of projectiles in overheated anti-aircraft guns, consisting of a low order detonation or the projectile in the breech of the gun. In previous reports, data were reported for the effect of temperature on the more common military high-explosives.

"Explosion" temperatures were determined for Composition A (R2431), pressed at 10,000 p.s.i., using the methods and apparatus previously described. The data are tabulated below (Table I), and heating curves for the experiments are shown in Figure 1. Composition A behaved quite similarly to RDX, except that no melting point break was found in the heating curves; in each case, rapid decomposition started at about 200°C., and no appreciable decomposition was observed below this temperature. Damage to the cylinders containing the explosive was almost the same as for pure RDX, and typical damaged cylinders are shown in the accompanying photographs.

Fig. I

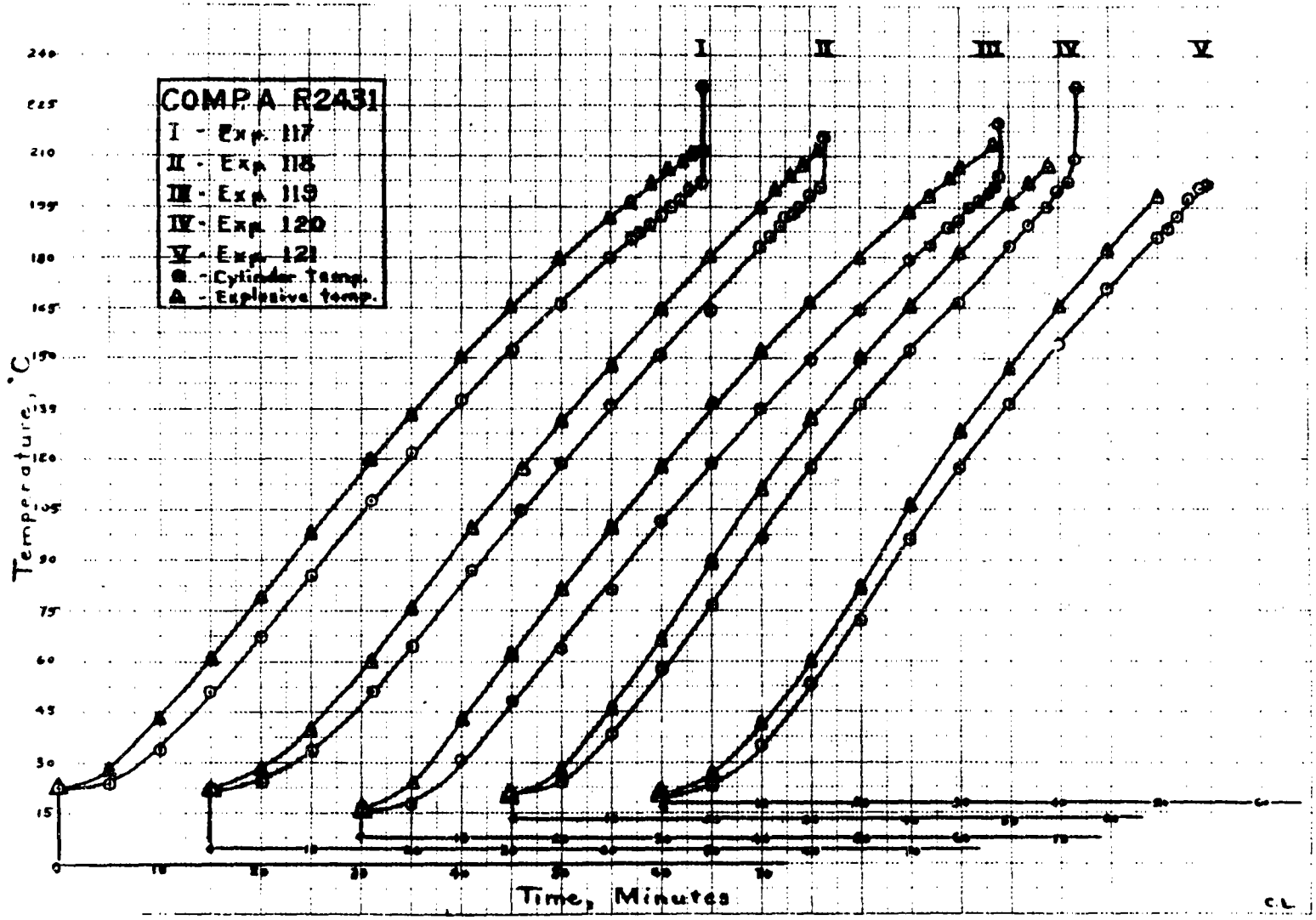
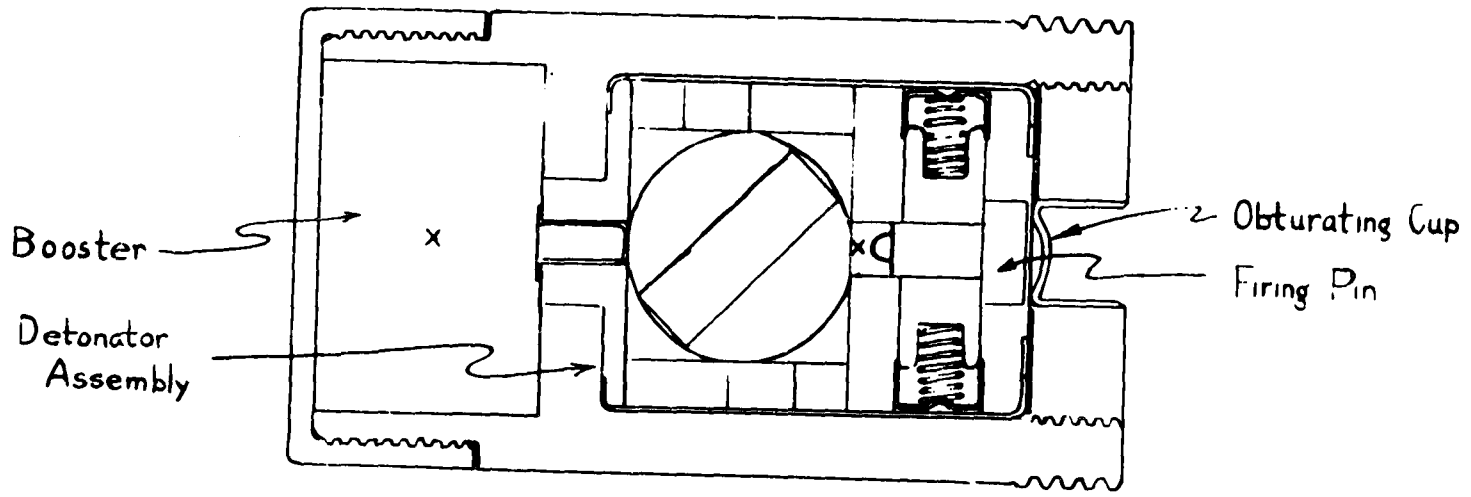

COMP A R2431
I - Exp. 117
II - Exp. 118
III - Exp. 119
IV - Exp. 120
V - Exp. 121
● - Cylinder temp.
▲ - Explosive temp.
I
II
III
IV
V
Temperature, °C
Time, Minutes

Table I

Exp. #	Charge Material	Charge wt. (gms)	Charge Density	Rate of heating (deg/min) above 100°C.	Time of explosion min.	Bomb temp. at exp. (°C.)	Final recorded value of charge temp. (°C.)	Lower limit of expl. temp. (°C.)	Upper limit of expl. temp. (°C.)	Action of explosion on bomb
117	Comp. A	10	1.5	2.9	64	213	230	170	202	Cylinder quartered
118	"	"	"	3.3	61	213	215	168	201	Cylinder split in half
119	"	"	"	2.9	64	214	220	-	204	Cylinder split in 3 pieces
120	"	"	"	3.3	57	213	230	175	205	"
121	"	"	"	3.6	55	212	201	-	201	Cylinder split in half

In the table, the lower explosion limit is taken as the point at which the rate of heating of the sample just exceeds the rate of heating of the bomb; decomposition of the explosive at an appreciable rate is beginning at this temperature. The upper explosion limit is taken as the point at which the internal rate of heating is twice the external rate; marked decomposition of the explosive is occurring at this temperature.

In connection with the specific problem of "cooking-off" in the case of Mark 35-3 five inch projectiles, experiments were carried out to determine the explosive material present which is least thermally stable. As used, these projectiles are loaded with Ammonium Picrate (Explosive D) pressed to a density of about 1.5 and equipped with a Mark 48 auxiliary detonating fuze in the nose and a Mark 28-8 base fuze. The Mark 48 fuze contains about 15 g. of Tetryl as booster, with mercury fulminate as initiator; the Mark 28-8 fuze contains about 5 g. of Tetryl as booster, with mercury fulminate as initiator. The former is a time fuze, the latter a contact fuze. Figure 8 shows a sketch of the Mark 46 fuze, and the various parts are shown in the accompanying photograph.



Sketch of Auxiliary Detonating Fuze Mk 46

x - thermocouple junctions

Fig II

In order to determine the damage done by detonation of a Mark 46 fuze, the detonator assembly was replaced by a mild steel plug and the booster detonated with a #6 detonator. The fragments obtained are shown in the photograph with the dis-assembled fuze. In studying the effect of temperature on the fuzes, experiments were made using complete fuzes, fuzes with detonator assembly removed, and with booster removed. The complete fuzes were arranged with a thermocouple inserted into the center of the booster through the bottom and sealed in place with porcelain cement; a second thermocouple was inserted in the firing pin well after removal of the pin, so that its junction rested on the case of the detonator and was sealed in the top plug with porcelain cement. In this way the fuze was maintained as nearly gas-tight as in its initial state. The data obtained with the complete fuzes are tabulated below (Table II), and photographs of the damage from the explosion are shown for two typical cases. Heating curves are shown for three experiments in Figure 3.

Table II

Ex. No.	Booster temp. at explosion °C.	Detonator temp. at explosion °C.	Rate of heating above 100°C. (°/min)	Time of explosion (min)	Action of explosion
6	146	---	1.3	77	Booster case fragmented
9	135	144	1.7	62	" " "
13	145	153	2.6	48	" " "
14	147	---	2.0	52	Fuze case fragmented
15	154	145	3.2	37	" " "
16	151	156	4.2	35	Booster case fragmented
23	143	165	6.5	26	" " "

In none of the above experiments was any sudden rise observed in the booster temp. prior to explosion, as was found in previous experiments with Tetryl when decomposition occurred.

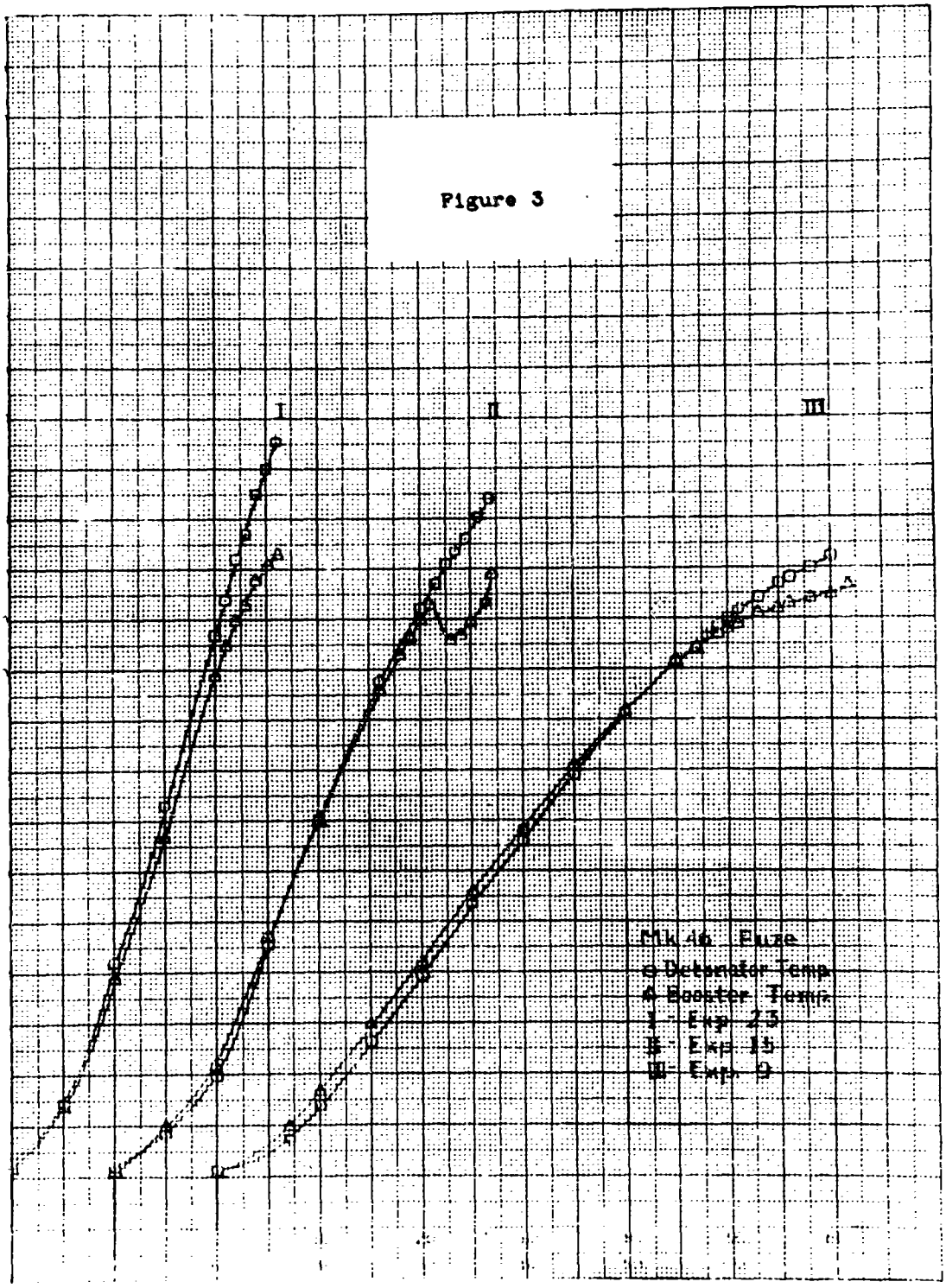


Figure 3
I
II
III
Mk 46 Fuze
Detonator Temp.
Booster Temp.
I - Exp. 23
II - Exp. 15
III - Exp. 9

Two experiments were run with the detonator assembly removed and a thermocouple sealed into the booster. The heating curve (Figure 4) is almost identical with that of Tetryl in previous experiments. In each case rapid decomposition started at about 175°C. and the characteristic break at the melting point (about 130°C.) was observed.

Several experiments were made with detonator assemblies and without the fuze case. In all experiments, the thermocouple was sealed in the firing-pin well. It was found that the explosion temperature rose with increased rate of heating, and a series of heating curves at various rates is shown in Figure 5. Part of this increase may be due to a greater lag in temperature between the point at which the temperature was recorded and the detonator material, but it is probably largely due to a real increase, as has been reported in an other investigation(1). The results are given in Table III.

Table III

Ex. No.	Explosion temp., °C.	Rate of heating above 100°, °/min.	Time of expl. min.
2	150	2.4	40
17	144	1.6	60
18	168	4.0	33
19	176	6.2	27
20	178	7.0	25
21	147	2.0	50
22	142	1.0	105

All the above experiments were run without the fuze case and the damage was identical; the case was torn and the inner parts thrown out, but no fragmentation occurred.

In order to determine the effect of the detonator explosion in the fuze case, three experiments were run with fuzes having the booster case filled with lead and a thermocouple inserted into this as in a regular booster. No damage was done to the fuze case and the only evidence of decomposition was a slight temperature rise (see Figure 6) between 145-150°C.

In order to further investigate the stability of the detonators, experiments were run in which fuzes, arranged as previously described were heated rapidly to some temperature below the

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(1) Tamman and Kroger, Zeit. Anorg. Chem. 169 1 (1928)

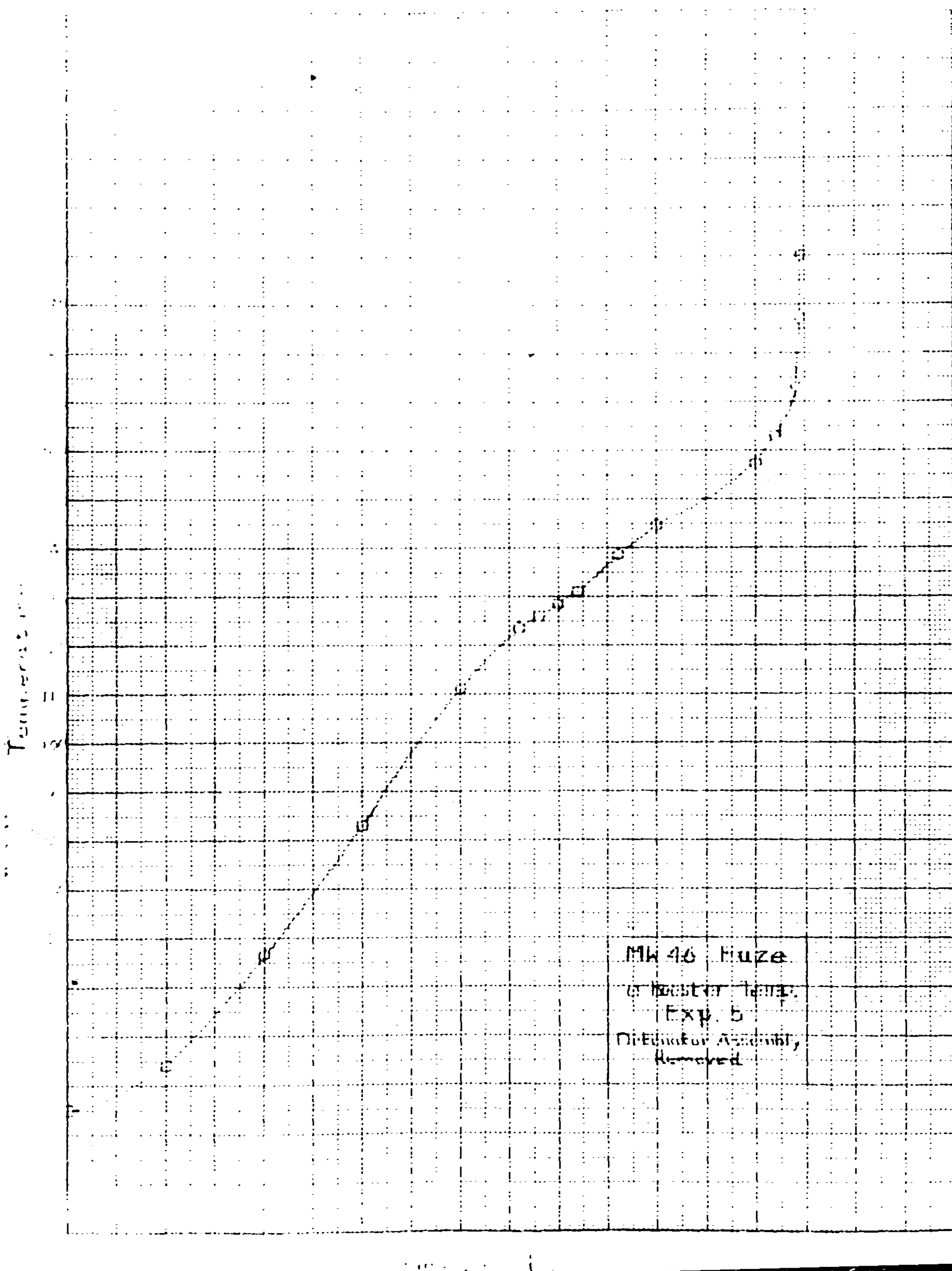


Fig 11

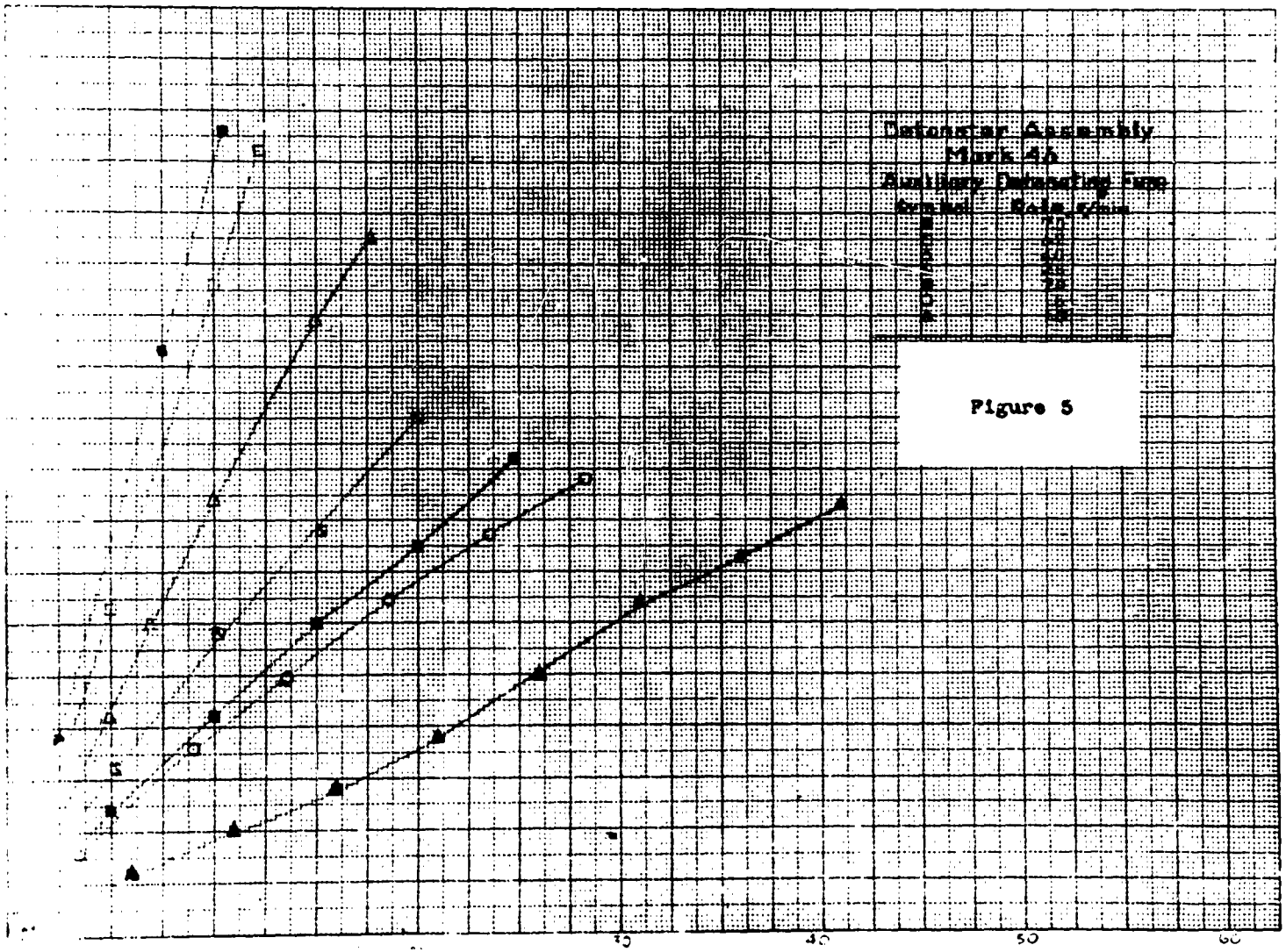

Detonator Assembly
Mark 4A
Auxiliary Detonating Fuse
Symbol
40
50
60

Figure 5

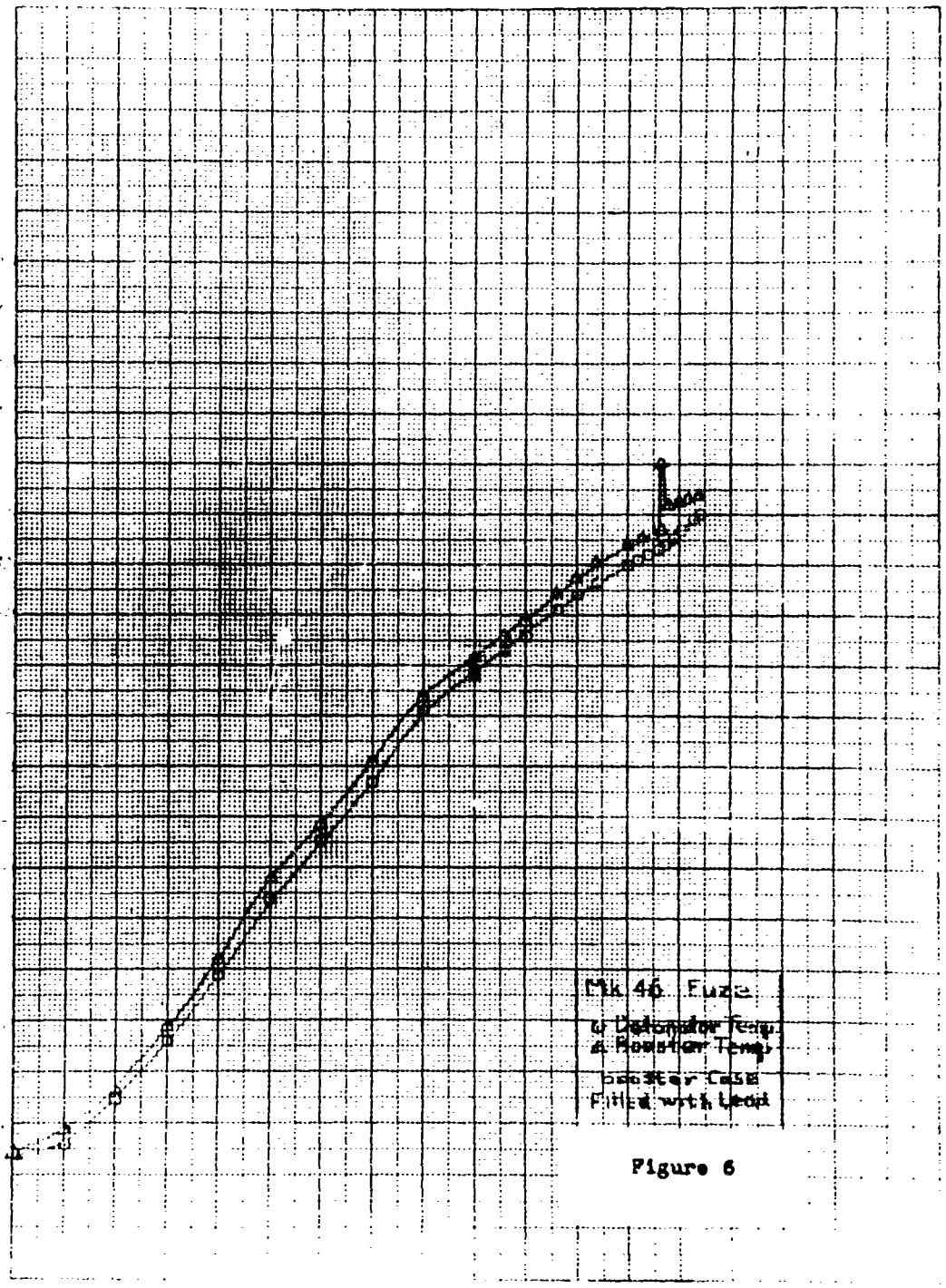


Figure 6

explosion temperature found at the slowest rate of heating, and held until explosion occurred. The results are given in Table IV.

Table IV

Exp. No.	Booster temp. °C.	Detonator temp. °C.	Time at temp. min.	Time to temp. min.	Results
24	133±1	135±1	15	23	Complete fragmentation
26	131±2	135±2	17	22	Booster case fragmented
25	127±2	127±2	55	50	Bottom blown off booster case
27	125±1	130±1	32	30	Complete fragmentation
28	122±2	122±2	180	25	No explosion

In Exp. 28, after being held at 122° for 3 hours, the temperatures were raised, and a normal Tetryl decomposition found, indicating that the detonator material had decomposed slowly during the heating. No temperature rise occurred before the explosion in any of the experiments.

Previous experiments indicate that Explosive D does not begin to decompose until a temperature of about 275°C, and that Tetryl, although decomposition starts at the melting point, has an upper explosion limit of 165 - 170°C. The present experiments show that the least stable point in the projectile is the fuze, and that the detonator is the least stable part of the fuze. The violence of the explosion of the fuze would probably be sufficient to at least cause a low order detonation of the main charge, which is what happened in reported cases of "cooking-off."

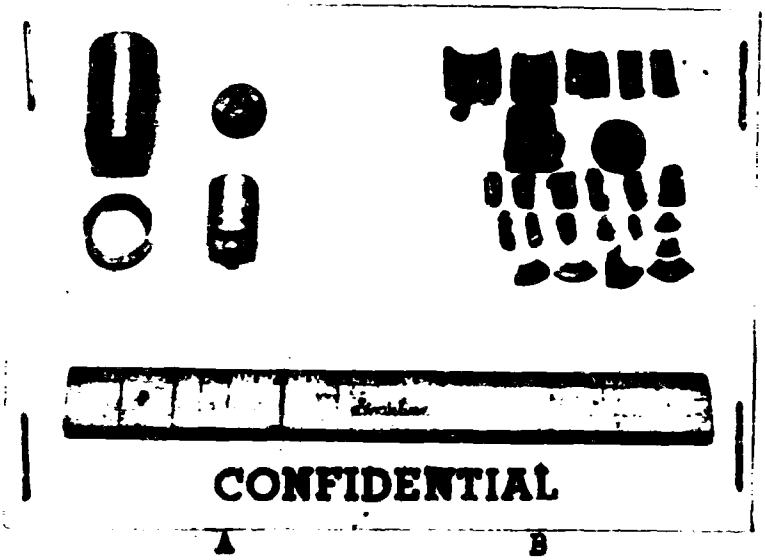
No experiments have been run with the Mark 28-3 fuzes, as it is felt that their behavior would be similar to the Mark 46, and it has been found difficult to insert thermocouples so that any very reliable temperatures can be obtained.

[redacted]

CONFIDENTIAL

A B

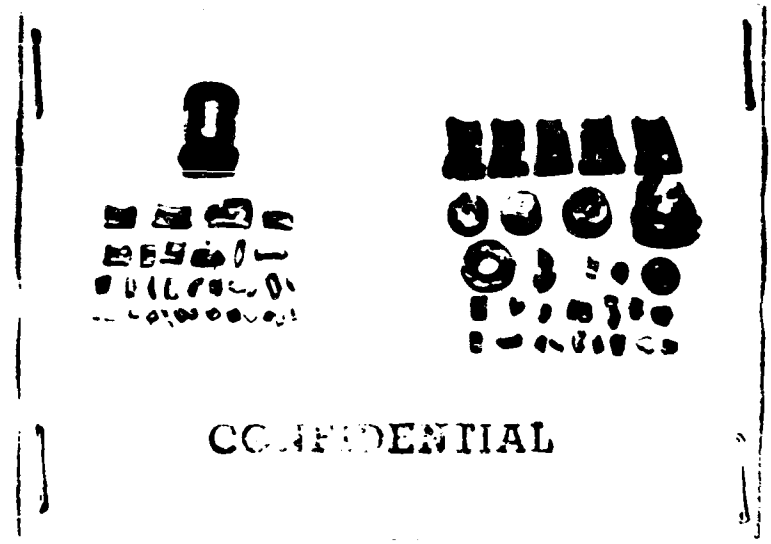
Comp. A A - Exp. 120 B - Exp. 121



A B

Mark 46 Auxiliary Detonating Fuze
A- Fuze parts (top, fuze case and top plug; bottom, booster cup and detonator assembly)
B- Fuze case after detonation

(mild steel plug in place of detonator assembly.)



A B

Mark 46 Auxiliary Detonating Fuze
A- Exp. 9 B- Exp. 15

HIGH EXPLOSIVES SENSITIVENESS INVESTIGATION

Report Submitted by
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Hercules Powder Company
Wilmington, Delaware

Sensitiveness to Impact

The object of this particular phase of the work on the sensitiveness of explosives is to determine the effect of sand-blasted surfaces on the impact sensitivity of PETN. It is desirable to have information on this subject because sand-blasted surfaces are especially useful in testing explosives such as Tetryl, wax desensitized PETN, etc.

In an early report*, W. S. Koski investigated the use of sand-blasted striking surfaces and found that the results obtained using 20 mg. of PETN and a 2 kg. weight were appreciably the same as those obtained with smooth surfaces. This conclusion was confirmed using No. 30 Corundum (aluminum oxide) as the sand-blasting medium and appears to hold true when Ottawa Flint Shot is used. However, when a coarser abrasive, No. 10 Corundum, is employed, this is not the case as the height of fall necessary to produce 50% shots is greatly reduced to a point approximately 3/5 of that necessary for smooth surfaces (see Table 2 and Figure 1). This relationship has been found to exist for amounts up to 40 mg., however, for amounts exceeding this, there is reason to believe that the height of fall using No. 10 Corundum approaches and is equivalent to the height of fall using the finer abrasives and smooth surfaces. It is planned to do future work using amounts in excess of 40 mg.

When amounts smaller than 20 mg. are used the height of fall for 50% shots deviates from the linear relationship. When 2.5 mg. is used the height is approximately 1/2 the height if the linear relationship existed for small amounts.

Future work will be done to determine the height of fall for small amounts of PETN using smooth surfaces.

Experimental, Procedure and Results

The Bureau of Mines Impact Machine, which was used for this work, has been described in the opening report,

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* Report dated 9-4-42, RI 2072, Hercules Powder Company.

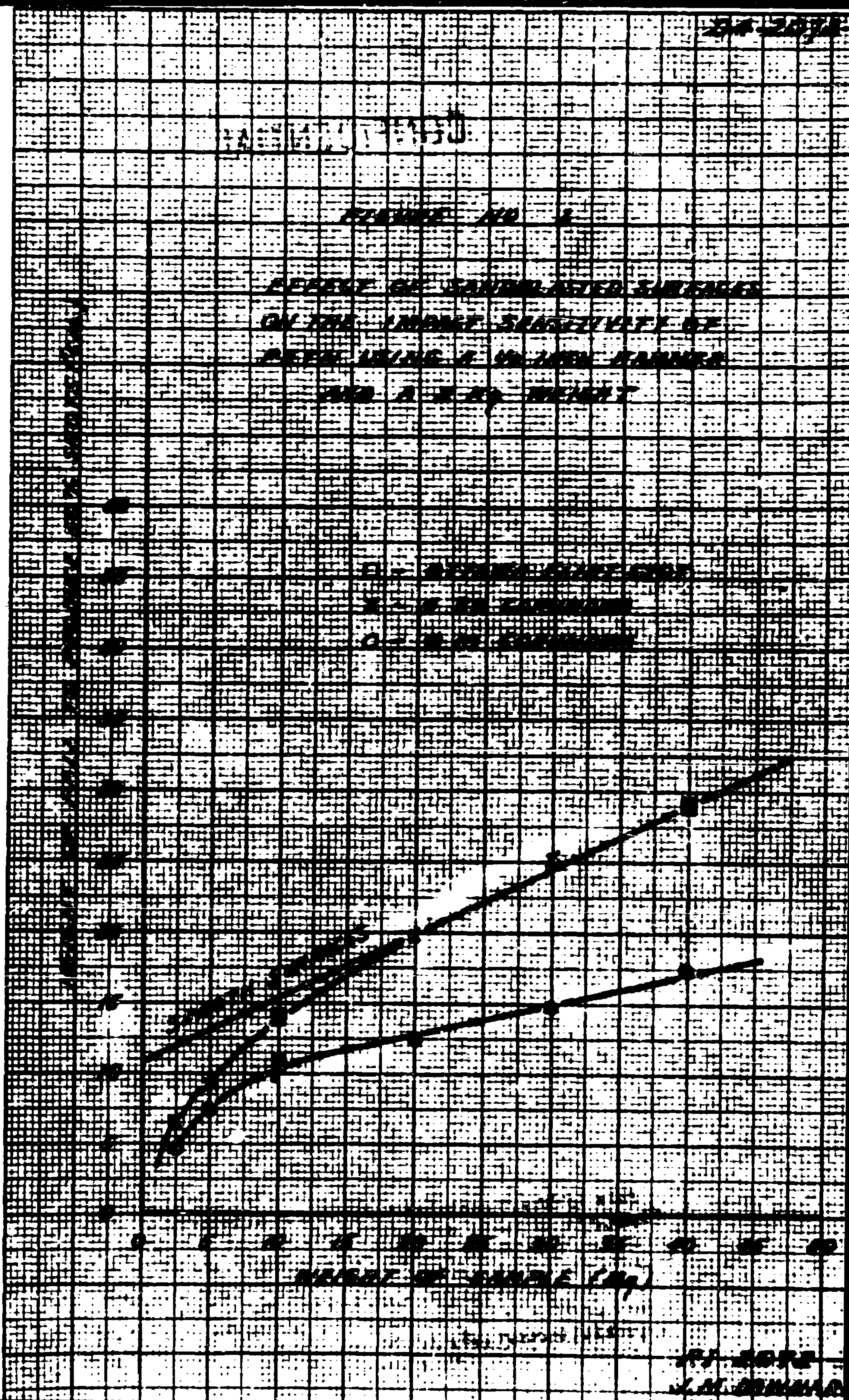


FIGURE NO. 1
EFFECT OF SANDBLASTED SURFACES
ON THE IMPACT SENSITIVITY OF

The hammers and anvils used were made of hardened tool steel and sand-blasted with various abrasives using a gun with an air jet of 7/32 in. and approximately 65-70 p.s.i. air. The three abrasives employed along with their particle size and surface roughness produced are listed in Table 1.

Table 1

Abrasive	Source	Average Particle Size (mm.)	Surface Roughness* (microns)
Ottawa Flint Shot	Ottawa Silica Sand Company	0.5	11
No. 30 Corundum	Abrasive Products Company	0.6	25
No. 10 Corundum	Abrasive Products Company	2.5	30

* The Surface Roughness is a measurement of the vertical distance between the high and low points on the surface.

The horizontal distances between the high or low points on the surfaces sand-blasted with No. 10 Corundum were about 5 or 10 times greater than those sand-blasted with No. 30 Corundum.

The PETN used was of the Standard Ensign-Bickford Primacord type and had been thoroughly dried. It had a screen test analysis as follows:

5.60% on 40 mesh
39.25% on 100 mesh
55.15% through 100 mesh

The RRL Method for determining the point for 50% shots was used. This method is described in full in the September 4, 1942 report by W. S. Koski.

In running the tests a practice was made of changing the hammer and anvil at least twice a run. If, at any time, there was a rapid change in the results, and these appeared to be out of line with previous data obtained, then the hammer and anvil were replaced. After each trial the hammer and anvil were cleaned off with acetone.

Table 2

Effect of Sand-Blasted Surfaces on the Impact Sensitivity of PETN Using 1/2 Inch Hammer and 2 Kg. Weight

Type Surfaces	Runs	Height of Fall (cm.) for 50% Shots					
		2.5 mg.	5 mg.	10 mg.	20 mg.	30 mg.	40 mg.
Ottawa Flint Shot	1	-	-	11.5	-	-	25.0
	2	-	-	16.0	-	-	33.0
	Av.	-	-	14.0	-	-	29.0
No. 30 Corundum	1	6.0	10.0	13.0	20.0	25.5	29
	2	7.0	8.0	15.0	22.0	25.0	29.5
	3	-	-	-	17.5	-	-
	Av.	6.5	9.0	14.0	20.0	25.5	29.0
No. 10 Corundum	1	4.0	8.0	11.5	13.1	15.0	16.0
	2	5.5	6.5	10.5	12.0	14.5	19.0
	3	-	10.5	-	-	-	-
	Av.	5.0	8.5	11.0	12.6	16.0	17.5

Sensitiveness to Friction

In the preceding report on this investigation, evidence was presented which indicated that for Cyclonite through 325 mesh the extrapolated height of fall for infinite pendulum weight was zero. This conclusion was most definitely shown by the experiments made with the roughest surfaces. It seemed to be of interest to find out whether this result also held for different particle sizes and different explosives. Accordingly, tests have been run on several explosives using fractions of various particle sizes and the roughest surfaces. This report gives the results obtained.

Results and Discussion

Effect of Pendulum Weight and Particle Size for Several Explosives

The variation of height of fall to produce 50% shots with pendulum weight has been studied for through 325 mesh and 50 or 60/100 mesh fractions of PETN, Tetryl, Cyclonite, and Haleite. The tests were made on hardened tool steel surfaces with a roughness of 30 microns: wheels 2 in. in diameter and 1/8 in. thick were used, the amount of explosive was 20 mg./cm^2 or more, and the angle of incidence was 4 deg. Weights of 3, 6, 10, and 20 kg. were added to the pendulum. The results are given in Table 1 and Figure 1.

The results for Cyclonite through 325 mesh were taken from the preceding report. In the case of Pentolite the shots were not too audible so the results there are not so certain as for the others. In all the other cases the shots were distinct. Even Tetryl, which cannot be heard when the wheels are used with smooth surfaces, gave shots which while not loud were quite definite.

In Figure 1 the heights of fall for the 50 or 60/100 and the through 325 mesh fractions of PETN, Cyclonite, Tetryl, and Haleite are plotted against the reciprocal of the pendulum weight. In all cases the extrapolated height of fall at infinite weight seems to be zero. This is in agreement with the results reported last month where the same behavior was shown by Cyclonite through 325 mesh with various strikers and values of the surface roughness.

The curves for Haleite are interesting in that they cross those for Tetryl and possibly also Cyclonite. In the region of heavy weights the order of decreasing sensitiveness is PETN, Cyclonite, Tetryl, Haleite. In the region of light weights it is PETN, Cyclonite, Haleite, Tetryl.

Figure 1 D4-2072-69

Variation of Friction Sensitiveness of Several Explosives with Pendulum Weight and Particle Size

Hardened Steel Anvils and 2" x 1/8" Wheels

Roughness = 30 μ
Amount = about 20 mg./cm.
Angle of Incidence = 4 deg.

- Tetryl 50/100 mesh
- Tetryl thru 325 mesh
- EDNA 60/100 mesh
- EDNA thru 325 mesh
- Cyclonite 50/100 mesh
- Cyclonite thru 325 mesh
- PETN 60/100 mesh
- PETN thru 325 mesh

Height of Fall to Produce 50% Shots in cm.

30
25
20
15
10
5

0.05 0.10 0.15 0.20

Reciprocal of Pendulum Weight in kg.

HERCULES POWDER CO.
R.I. 2072 4-10-44
J. G. Fox

In all cases, the small particle size is more sensitive than the large. This is in agreement with all past results on the effect of particle size on the sensitiveness to frictional impact.

The heights of fall for regular Cyclonite, Tetryl, Haleite, Pentolite, and Pentryl* have also been measured. The values for the regular particle size are, in general, intermediate between those for the coarse and fine particle sizes as is to be expected. They have not been plotted in Figure 1 in order to avoid making the graph too complicated. However, graphs of the heights of fall of Pentolite and Pentryl show that they too extrapolate to zero height at infinite weight.

The sensitiveness of Pentryl seems to be very nearly the same as that of Cyclonite over the whole range of weight. Pentolite and Tetryl seem to have approximately the same sensitiveness throughout, although there is some evidence that for light weights Tetryl is less sensitive.

The decrease of height of fall with increasing pendulum weight is of interest as indicating a possible means of extending the range of the apparatus to include the less sensitive explosives. The heights of fall at 10 and especially 20 kg. are so low as to lead one to believe that the heights of fall of explosives as insensitive as TNT would, under the same conditions, be less than 50 cm. the limit of the present apparatus. Several such explosives have been tried; namely, TNT, Picric Acid, Ammonium Picrate, and Nitroguanidine. In no case were shots definitely audible. It is felt, however, that this was due more to the small amounts of explosive shooting possibly because of low propagation sensitiveness, than to insufficient height of fall for initiation. A striker designed to have a larger area of contact will be tried in an effort to overcome this difficulty. In trying this, however, one will run into the difficulty that the height of fall increases for strikers with larger areas of contact. Areas of contact large enough to give audible shots with these explosives may require heights of fall greater than are possible with the present apparatus.

Future Work

Tests will be made on a few initiating explosives to see where they fall in the order of sensitiveness. A striker made in the shape of a segment of a sphere with a large radius will be tried in an attempt to make the less sensitive explosives shoot.

- -

* (2,4,6-trinitrophenylnitramino) ethyl nitrate

Experimental

The sources of the explosives used were as follows:

PETN - received from Kenvil Plant 11/1/43
Cyclonite - RDX, ¾., R-2106, received from Bruceton 11/3/43
Tetryl - form used in blasting caps, obtained from duPont
Haleite - SW 96, obtained from duPont
Pentolite - made in the laboratory, analysis - PETN/TNT 49.2/50.3 with 0.5% volatile
Pentryl (2,4,6-trinitrophenylnitroamioethyl nitrate) - made in the laboratory about ten years ago by Mr. W. deC. Crater (RI 3519, October 6, 1933).

The screen analysis of the PETN, Cyclonite, Haleite, and Tetryl used is as follows:

	Tetryl	PETN	Cyclonite	Haleite (SW 80)*
On 50 mesh	4%		1%	46%
On 40 mesh		6%		
On 100 mesh	5	39	8	51
Through 100 mesh		55		
On 200 mesh	40		44	2.7
Through 200 mesh	51		47	2.3

* There was no more Haleite SW 96 remaining at the time the screen analysis was made so lot SW 80 was analyzed. The sensitiveness of Haleite SW 80 and SW 96 had previously been found to be the same.

Table 1

Variation of Sensitiveness to Frictional Impact of Several Explosives with Pendulum Weight

Anvils - hardened Stentor steel
Wheels - hardened Stentor steel, 2 in. dia., 1/2 in. thick
Surfaces - sand-blasted with No. 10 corundum, roughness - 30 microns
Amount - about 20 mg.
Angle of Incidence - 4 deg.

Height of Fall to Produce 50% Shots in cm.

Weight added to Pendulum	1/M*	PETN Thru 325	PETN Reg.	PETN 60/100	Cyclonite Thru 325	Cyclonite Reg.	Cyclonite 50/100	Haleite Thru 325	Haleite Reg.	Haleite 60/100	Tetryl Thru 325	Tetryl Reg.	Tetryl 50/100	Pentryl Reg.	Pentolitese** Reg.
3 kg.	0.21	7.6		10	11	13	18	17	16	18	24	24	30	13	21
6	0.13	3.9		3.6	7.3	10	12	14	12	17	16	15	16	9	15
10	0.086	2.6		2.2	4.9	6.5	7.6	10	8.7	12	10	9	11	7.1	13
20	0.046				2.3	4.0	4.1	5.6	5.9	7.1	5	5.7	6.0	3.9	5.2

* M is the sum of the figure in column 1 and 1.7 kg., the weight of the pendulum alone.

** The shots with Pentolite were not very audible so these results are subject to some uncertainty.

Sensitiveness to Glancing Impact

Sliding Rod Tests

Sliding Rod Design and Operation

The sliding rod impact machine (see PT-18, page 28 ff.) was introduced into the investigation of high explosive sensitiveness so that a study could be made of the "glancing" action produced by the device upon military explosives. An adequate explanation of the operational procedure and a description of the apparatus can be found in the earlier report.

Past work was concerned with obtaining the necessary standardizations for making reproducibility of results possible with this instrument. An investigation was made of the effect of amount on the sensitivity of PETN at 30°, 45°, 60°, and 75° slide angle.

Recent work has involved a study of the effect of various metals on the sensitivity of PETN. Different anvil surfaces varying in composition and hardness have been employed as test pieces in a classification of their relative sensitivity (or safety value) when using a standardized sample of PETN.

Sliding Rod Tests

Recent results and previous data have indicated that it is very necessary to use the following standardizations if reproducibility of results is desired:

1. Screened fraction of explosive.
2. Weight of sample in the range not affected by amount.
3. Sand-blasted surfaces (producing uniformity).

The PETN used exclusively in these experiments was screened through 40 mesh and retained on 100 mesh. A 38-40 mg. sample weight was used because earlier tests showed little effect due to amount in this range for all angles. As before, preliminary tests again showed lack of reproducibility of data if an attempt was made to employ smooth machined anvil surfaces. The anvil faces were sand-blasted, but it was not necessary to roughen the hardened steel head also since the results were equally reproducible with smooth or roughened heads.

Three intermediate slide angles were added to determine more closely the actual curves of the plotted data: 37.5°, 52.5°, 67.5° added to 30°, 45°, 60°, and 75°. The plots for various metallic surfaces of the heights of fall producing 50% detonations of PETN versus the slide angles are closely "U-shaped". The vertices or minimum limiting values are in the 60°-67.5° slide angle range. Hence, the position at which most metals effect the greatest sensitivity of PETN on the sliding rod is between a 60° and a 67.5° angle of incidence of rod and anvil.

Very little evidence was found to confirm the view that the hardness values of the metals are in direct relationship with their glancing impact sensitivity and this property cannot be used for predeterminations. Some relationship does exist but it is not apparent at this time.

A few metals such as copper and phosphor bronze exhibited a sharp rise in the curves of their plotted values in the vicinity of 60°. This produced a secondary vertex showing a definite desensitization at the 60° angle. No attempt will be made to explain this unusual feature. However, the change definitely does not fall within the range of experimental error.

Future Study

Future work with the sliding rod will be concerned with a study of various high explosives. Their range of sensitiveness, the individual effect of amount and particle size will be investigated and a definite classification will be made. An attempt will be made to correlate the glancing impact data with results obtained on the direct and frictional impact-producing devices.

Discussion of Results

A study has been made of the effect on the sensitivity of PETN of various metallic surfaces. Different metal anvils have been employed on the sliding rod machine in this testing while using a hardened steel striker throughout. From the results of previous work on this machine, it was found necessary to use a screened fraction of PETN and at least a 30 mg. sample weight. By having these standardizations, the anomalous results caused by the effect of amount and particle size were avoided. A 38 to 40 mg. sample of PETN screened through 40 mesh and retained on 100 mesh was used as standard.

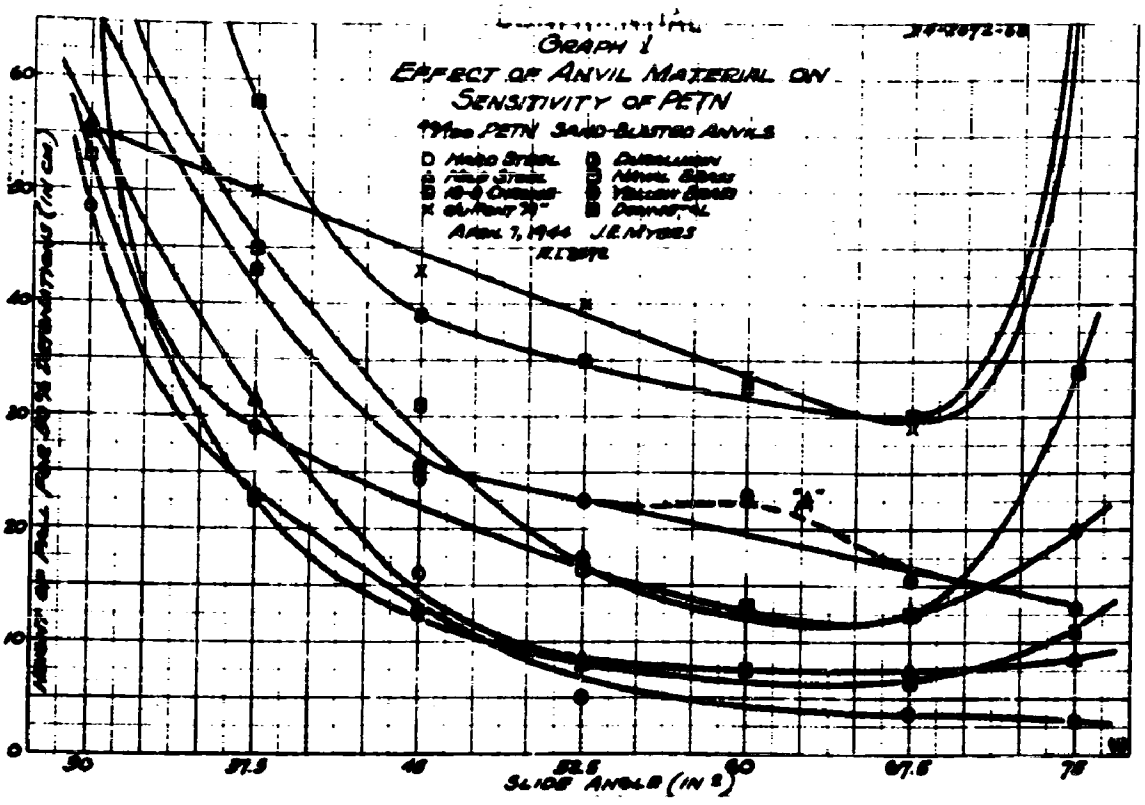
Preliminary work on smooth machined anvil surfaces, supposedly presenting uniformity, indicated (as was shown in the earlier report) that good reproducibility of results was not possible under these conditions. See results shown in Table 1. However, data could be obtained under better circumstances when the anvil surfaces were roughened by sand-blasting which produces a uniform surface. It was not necessary also to roughen the rod nosepiece. The values were somewhat higher than those found with both surfaces blasted but were easily reproducible in most cases.

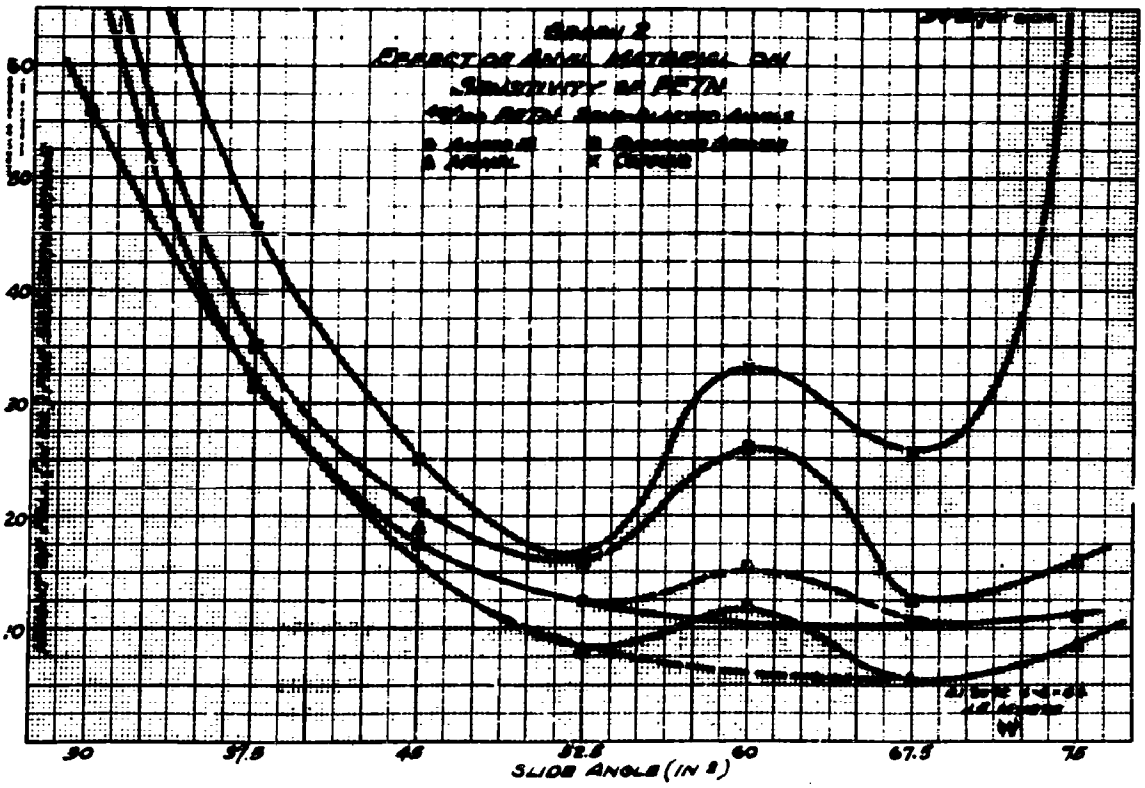
Fourteen metals of various hardness and composition have been investigated. At first the slide angles used in testing the surfaces were the original 30°, 45°, 60°, and 75°. Upon examining the plot of the height values versus the slide angle, it was evident that intermediate angles should be studied to determine the correct curve and vertex. This led to the adoption of the three following intermediate slide angles: 37.5°, 52.5°, and 67.5°. The final results using sand-blasted surfaces, intermediate angles, and sized PETN are given in Table 2. Upon plotting these averaged values (height of fall for 50% detonations versus slide angle), Graphs 1 and 2 are produced. The majority of the metals tested have been included in Graph 1. Those exhibiting an unusual curve are found in Graph 2.

Graph 1: The general shape of the curves resembles a large "U" with the vertex (or minimum height) in the region between 60° and 67.5° slide angle. Metals which fail to cause PETN to detonate when struck by the rod from angles of 30° or 75° become very sensitive if the angle is changed toward 60°. Hence the height at which detonation will take place on a metal anvil is very dependent upon the slide angle or the "angle of incidence" of the striker and the surface.

At 75° there is a definite upward swing to many of the curves which would follow the direct impact data on various materials. At 90° the softer metal surfaces fail to produce detonation at heights above those found at 75° on the sliding rod. There is not much evidence available from these data which indicates that the hardness values can be used as a direct measurement of the sensitivity. However, the results do appear to be related in some manner to the respective hardnesses.

Graph 2: From the plot of data on yellow brass anvils from Graph 1, it is possible that the alternate dotted curve, "A", could be chosen. This would produce a slight rise in the original curve, the highest point near 60° slide angle. The four metals plotted on Graph 2 very definitely indicate this


GRAPH 1
EFFECT OF ANVIL MATERIAL ON SENSITIVITY OF PETN
APRIL 7, 1944 J.E. MYERS
SLIDE ANGLE (IN °)
30
37.5
45
52.5
60
67.5
75


SLIDE ANGLE (IN °)
30
37.5
45
52.5
60
67.5
75

reverse curve and second vertex near 60°. The change is slight for monel and Ampco 18 and alternate optional curves have been included. For copper and phosphor bronze this unusual effect is so definitely apparent that it could not be classified as a conceivable experimental error. At least eight determinations have been made for each of the three points in question: 52.5°, 60°, and 67.5°. No explanation is offered for this effect at this time.

During these series of tests it became apparent that it was possible to "work-harden" copper anvils easily after they had been used once or twice. This property of copper was also noticed in alloys tested that contained some copper. Necessary precautions were taken in using these copper-bearing anvils so that the resultant values were not changed by the change in hardness due to the impacts.

Summary

1. When using various roughened metallic surfaces as anvils there is produced a minimum or limiting value for height of fall necessary to cause 50% detonations of PETN in the vicinity of 60 to 67.5° slide angle. This effect becomes less pronounced as the anvil hardness is increased.

2. The Rockwell static hardness test does not influence the sliding rod results as much as would be expected. Usually, materials appear to have good safety properties (failing to detonate sensitive explosives) if they are appreciably deformed by the impact of a striking weight. The prime examples of this characteristic would be lead or aluminum. If the material is relatively plastic (elastic) the energy of the percussion is absorbed by the material and not transmitted directly to the explosive particles resting on its surface. It was apparent at first that the static hardness of the anvil had an important effect on the sensitivity (or safety) of the material. However, later tests have indicated some other factors are at least as important. Since these recent tests would necessarily disprove the relationship between hardness and sensitivity, it has been proposed that the "coefficient of plasticity" might possibly be adopted as a standard measure of sensitivity. This proposal would, of course, involve considerable experimental work to determine the advantage of classifying materials under this system.

Experimental

A. Materials

1. Explosives: Regular Primacord-Bickford type PETN has again been used throughout these tests in a special screened

fraction: "through 40 mesh, on 100 mesh". This was chosen as the standard fraction as mentioned in the earlier report.

2. Anvils: Table 3 is a list and description of those metals that were studied as anvil surfaces during recent work. The pieces were machined either into 1 in. x 1 in. x 1/4 in. squares or 1 in. diameter x 1/4 in. discs. Some preliminary work was made using machined surfaces. The major part of this study was done using sand-blasted surfaces. The roughened surfaces were made by blasting with Ottawa Flint Shot sand (1/2 mm. average particle size), air pressure: 65 to 70 p.s.i., air jet: 7/32 in. diameter.

3. Nosepieces: The removable head or nosepiece of the sliding rod used was a hardened Stentor tool steel striker with a machined surface (Rockwell Hardness: 60 "C" scale).

B. Apparatus and Procedure

The testing outline and description of the sliding rod machine can be found in PT-18, page 28 ff.

Table 1

Effect of Anvil Material Variation on Sensitivity of PETN

(Preliminary work using smooth anvil surfaces)

10 lb. Rod weight 40/100 PETN 38-40 mg. sample
Hardened steel machined head, 60 "C" Rockwell Machined Anvils

Perpendicular Height of Fall (cm.) for 50% Detonations; at Slide Angles of:

Anvil Material	30°	45°	60°	75°
Phosphor Bronze	10F at 70 10F at 70	10F at 100 88, 87, 99	10F at 130 88 10F at 130	38% at 140 10F at 140
Monel	33% at 70 10F at 70	58, 60 88, 99	66, 77, 10F at 130 10F at 130	20% at 140 10F at 140
Duralumin	10F at 70	10F at 100	10F at 130	10F at 140
Hard Lead	10F at 70	10F at 100	10F at 130	10F at 140
Copper	10F at 70 10F at 70	10F at 100 24% at 100	10F at 130 15% at 130	10F at 140 10F at 140
18-8 Chrome	50% at 70 10F at 70	20, 18, 26, 26, 28	37, 39 20	64, 70 40

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Table 2

Effect of Variation of Anvil Material on Sensitivity of PETN

10 lb. Rod weight 40/100 PETN 38-40 mg. sample
Hardened steel machined head, 60 "C" Rockwell Sand-blasted anvils

Anvil Material	Rockwell Hardness	Perpendicular Height of Fall (cm.) for 50% Detonations at Slide Angles of:						
		30°	37.5°	45°	52.5°	60°	67.5°	75°
Hard Lead	69R	10F at 70	-	10F at 100	-	10F at 130	-	10F at 140
Aluminum	86M	10F at 70	-	86	-	105	-	10F at 140
Dow Metal/	16B	10F at 70	58	39	35	32.5	30	10F at 140
Copper*	42B	10F at 70	45.5	25	16	33	25.5	114
Yellow Brass/	60B	66.5	43	25.5	22.5	23	15.5	13
Naval Brass/	66B	75	45	31	16.5	13	12.5	34
Duralumin/	73B	10F at 70	29	24.5	17.5	13	12.5	20
Phosphor Bronze*	74B	80	35	21	16	26	12.5	16
Monel*	76B	75	31.5	16.5	8	12	5.5	8.5
duPont "A"/	81B	55	50	43	40	33.5	29	10F at 140
18-8 Chrome/	82B	53	22.5	12.5	8.0	7.5	6.5	11.0
Ampco-18*	(82B (2C	56	32	17.5	12.5	15.5	10.5	11.0
Mild Steel/	(93B (16C	56	31.5	14	8.5	7.5	7.5	8.5
Hard Steel/	60C	48.5	23	16	5	7.5	3.7	3.0

Increasing Hardness

/ Plotted on Graph 1

* Plotted on Graph 2

(All values average of 3-8 determinations)

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Table 3

Anvil Material

Anvil Material	Description or Composition (%)	Rockwell Hardness
Hard Lead	10 Sb; 90 Pb	6 9R
Aluminum	No. 2S 99.9 Al	86M
Dow Metal	No. O-1; 8-1/2 Al, 1/2 Zn; Mg	18B
Copper	99.9 Cu; electrolytic	42B
Yellow Brass	65 Cu; 35 Zn	60B
Naval Brass	60 Cu; 39.25 Zn; 0.75 Sn	66B
Duralumin	No. 24 ST 4.5 Cu; 0.6 Mn; 1.5 Mg; Al	73B
Phosphor Bronze	"A" - 95 Cu; 4.3-5 Sn; P	74B
Monel	67 Ni; 30 Cu; 1.4 Fe; 1.0 Mn; 0.1 Si	76B
duPont "A"	88 Cu; 10Sn; 2 Pb	81B
18-8 Chrome	Similar to 302 SS: 17.5-20 Cr; 8-10 Ni; 1.25 Mn; 0.08-0.2C	82B
Ampco 18	84.6 Cu; 16.3 Al; 3.7 Fe; 0.2 Ni	(85B (2C
Mild Steel	Cold Rolled Stock	(93B (16C
Hard Steel	Stentor Tool Steel	60C

Increasing Hardness ↓

Rockwell Scales

"M" 100 kg. major load, 1/4 in. ball penetrator, red scale.

"B" 100 kg. major load, 1/16 in. ball penetrator, red scale.

"C" 150 kg. major load, Braile penetrator, black scale.

"R" 60 kg. major load, 1/2 in. ball penetrator, red scale.

Sensitiveness to Rifle Bullet

Recent tests have been conducted on the pipe disc-bomb alterations which are a continuation of the series studied in an earlier report. The following variations affecting the explosive sensitiveness have been investigated:

1. Effect of container alterations with respect to disc, base, and casing thickness and/or material.

2. Classification of various cast explosive mixtures.

The procedure described in earlier reports for testing and preparing the bombs has been followed during this experimentation. This involves firing the bullet longitudinally into a container charged with explosive. This bomb consists of a short length of pipe to one end of which a steel plate has been welded. A steel frontal disc at which the bullet is fired is inserted in the open end to rest on the column of explosive. Current results have indicated the following generalizations. The classification of explosions called "failures" are known as either complete or partial detonations.

1. A definite decrease is apparent in the percentage detonations produced with cast Pentolite when using the standard disc-bomb construction (most effective arrangement) if the bomb diameter and the charge length are increased simultaneously.

3 in. charge, 1 in. diameter 80% failures; 62% complete
6 in. charge, 1 in. diameter 100% failures; 50% complete
8 in. charge, 1 in. diameter 80% failures; 75% complete
4 in. charge, 1-1/2 in. diameter 100% failures; 30% complete
6 in. charge, 1-1/2 in. diameter 70% failures; 14% complete
4 in. charge, 2 in. diameter 100% failures; 0% complete

2. Under standard disc-bomb construction the percentage detonations increases (when using cast Pentolite) as the hardness and/or the strength of the container material increases.

all steel (standard	100% failures
all monel	50% failures
all duralumin	20% failures
all copper	10% failures
all hard lead	0% failures

3. A slight desensitization is noticeable if wax is added to Torpex or Composition B cast mixtures when fired with standard disc-bomb container.

Torpex I (unwaxed	100% failures
Torpex II (1% beeswax)	70% failures
Composition B (unwaxed)	30-50% failures
Composition B (1% beeswax)	0-20% failures

4. If a nonmetallic liner is used as a secondary disc and inserted between the steel frontal disc and the explosive surface there is a definite decrease in the sensitiveness of cast Pentolite.

regular construction	100% failures
1/16 in. cellulose acetate secondary liner	30% failures
1/8 in. soft rubber secondary liner	10% failures
3/16 in. maple wood secondary liner	20% failures

5. Detonation originates solely by impact and penetration if the bullet is fired at the rear of the disc-bomb through the welded steel base plate into the Pentolite; and detonation produced by a similar penetration of the frontal disc is not due to frictional initiation against the side wall.

6. Various cast mixtures tested appear to be either slightly less sensitive than Pentolite or much less sensitive when fired at in the regular container.

50/50 Pentolite	100% failures	(80% complete)
Pentonal	100% failures	(20% complete)
Baronal	100% failures	(0% complete)
Minol II	100% failures	(0% complete)
DBX	90% failures	(0% complete)
50/50 Cyclotol	80% failures	(0% complete)
Tetrytol	50% failures	
TNT-Aluminum (85/15)	0% failures	

Future work will be confined to the following problems:

1. Extensive study on the desensitization effect of secondary disc liners and container coatings of plastic or resinous material.

2. Use of special experimental ammunition in comparing variations in container construction.

3. Comparison of the sensitivities of various cast and pressed explosive mixtures.

STUDIES OF THE PREPARATION, STABILIZATION AND TESTING OF EXPLOSIVES

Report Submitted by
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The Theory of Stability Tests

(H. Henkin)

Summary

The investigation of the theory of the adiabatic ignition test has been continued. A new apparatus has been constructed which, while still crude, is an improvement over the former one. With this apparatus, the ignition time for NENO (starting at 120°) was found to be 16 minutes. The prediction previously made was 13 minutes; the time found in the older apparatus was 20 minutes.

A beginning was made in the problem of testing smokeless powders. The equation for the time-temperature relationship in adiabatic ignition, derived in last month's report is

$$(1) \qquad t = \frac{1}{AZT_p^2} \left(T_o^2 e^{A/T_o} - T^2 e^{A/T}\right)$$

where T_o the initial temperature, T the temperature at time t, and A, Z and T_p are constants previously defined.[1]

One of the things which can be deduced from this equation is the following: If a series of adiabatic ignition tests is started at different initial temperatures, it should be possible to superimpose all of the time-temperature curves provided those obtained at higher temperatures are properly displaced along the time axis. The displacement of the time-temperature curve for any individual sample relative to that for another must be the time it takes for the sample started at the lower temperature to heat adiabatically to the higher starting temperatures.

A set of experiments to test this prediction was carried out with a sample of EJ powder (perchlorate filled double-base powder stabilized with Centralite). The results, shown in Figure 1, completely conform to the predictions from equation (1). This equation has been derived using the assumption that the

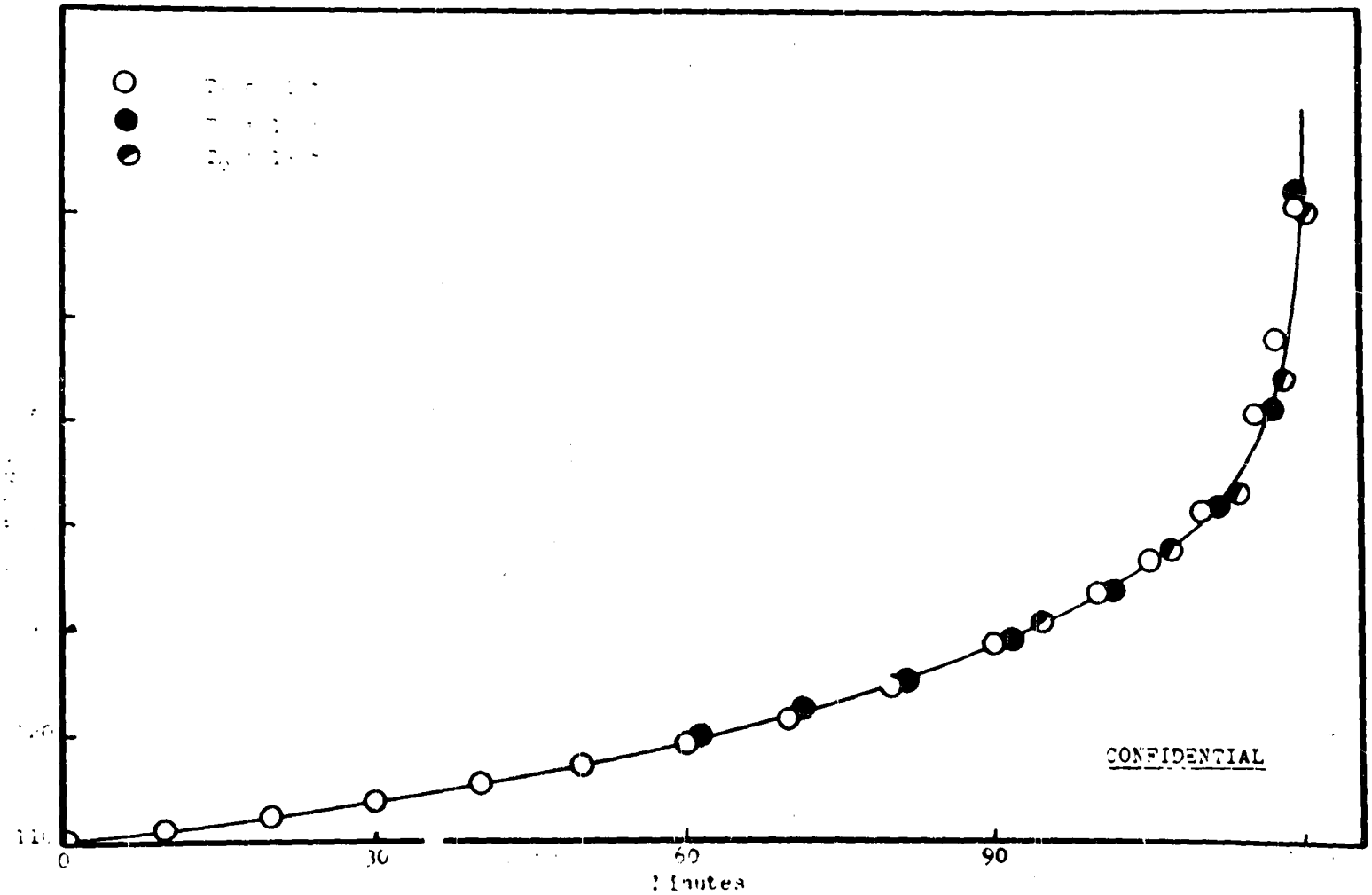

Figure 1 - ADIABATIC TEST OF DJ POWDER

increase in rate of decomposition with time is exclusively thermal in origin. It follows, therefore, that for the sample in question, and, at least, in the temperature range 110-130°C., the stabilizer has effectively prevented autocatalysis. The computation of the constants A, Z and T_0 for EJ powder has not yet been carried out. The smooth curve in Figure 1 is not, then, a theoretical one. It is further to be noted that the stability of EJ powder compares favorably by the adiabatic ignition test with the trench mortar powder previously tested by Hercules Powder Company[2].

Experimental

The new thermostat consists of an aluminum block with two wells drilled at positions equidistant from the center. In one of these a thermometer and thermocouple are placed. In the other well, which is 1-1/4" in diameter, the sample of powder is suspended. The "coefficient of thermal leakiness"[3], is defined as the constant K in equation (2)

$$(2) \qquad \frac{dt}{dt} = K \Delta T$$

where T is the temperature at time t and ΔT is the temperature difference between the thermostat and the sample of powder being tested. With our apparatus, this constant is 0.12 min.$^{-1}$. The temperature of the thermostat can be controlled at present to within ± 0.05°C. of that of the sample. It follows, therefore, that the maximum error which can be made in our present apparatus is 0.12 x 0.05 = 0.006°/min. (The actual error will be considerably smaller than this, since the error of 0.05°C. will not always be in the same direction.) With the present apparatus, any experiment can be carried out with reasonable accuracy provided that the sample decomposes fast enough so that it heats itself up (initially) at a rate which exceeds 0.06°/min. Further, it is possible to raise the temperature of the thermostat at a rate of about 10-15°C. per minute. This fast rate of heating permits the maintainance of adiabatic conditions almost until the ignition of the sample occurs.

Aluminized Explosives

(S.D. Brewer, J.W. Dawson, A. Turk)

In a previous British report[4], a method was set up to study the spewing of Minol. This method involved a measurement of the increase in volume of a 100 g. sample of Minol contained in a long tube. The assumption was implicit in this method that all the gas generated was held in the sample, and did not escape. This assumption is not valid. A better apparatus has been designed, and is shown in Figure 2. A charge of Minol is placed in the bulb of the apparatus, the standard joint well greased and held together with wire springs, and the whole device (almost up to the T seal) immersed in a steam bath at 100°C. The manometer is made of capillary tubing; there has been no evidence to date of water condensed in this capillary. The sample is heated in the steam bath for 15 minutes to attain equilibrium, the stopcock is then closed. The manometer is read at convenient intervals; the rise in pressure is of the order of 20-100 mm. in a few hours. This apparatus is accurate, easy to read, and measures the volume of gas which is evolved from the sample as well as that which is entrapped in it. Trial experiments showed that this is not even approximately true of the British apparatus. In practice, with a large mine, most of the gas which is generated will probably be trapped; the new apparatus therefore more nearly approximates actual service conditions.

With the aid of the new apparatus, a large number of experiments has been made. Variation in method of mixing, water content, method of preparation of the aluminum, etc., have been tested. Two different methods have been found which reduce the gas evolution by a factor of five to ten. The data are recorded in Table I and summarized in the following paragraph.

Minol containing a few tenths percent water when heated to 100°C. generates gas at a rate of about 5-8% of its own volume per hour. The exact amount of gas generated depends on the method of mixing the sample and the purity of constituents. The variations with method of mixing are quite complicated, but in general it can be stated that the greatest gas evolution occurs when water is added last to the mix. If all reagents are purified and carefully dried, gas evolution drops to 1-2% per hour of the volume of the mix.

Minol has been treated so as to reduce the gas generated despite the fact that commercial chemicals containing small amounts of water were used. One method of doing this consists of adding 4% anhydrous magnesium nitrate and 0.3% stearoxyacetic acid to

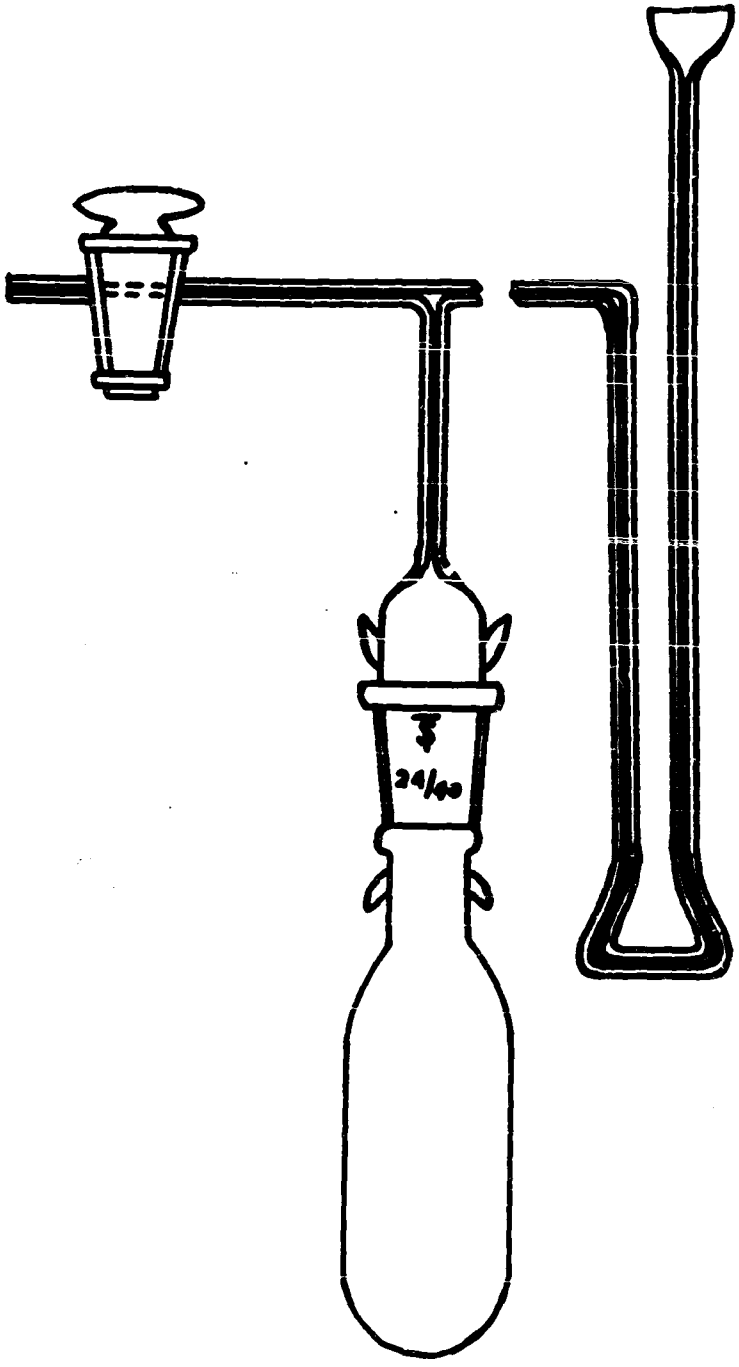

Figure 2 - Manometric Expansion Apparatus

the mix. Presumably the anhydrous magnesium nitrate combines with the water present; the stearoxyacetic acid prevents the separation of the mixture which otherwise occurs in the presence of magnesium nitrate. Alternatively, the aluminum can be treated with phosphate or silicate to produce a insoluble coating on the surface. The best phosphate coatings have been put on in fairly strongly acidic or basic solutions, and have cut the rate of gas evolution down to about 0.9% of the volume of the sample per hour.

In a typical case, 50 g. of aluminum powder was shaken intermittently for an hour at room temperature in 100 cc. of a 0.1 molar solution of phosphoric acid. The aluminum powder was then filtered, washed with water, and dried. The dried aluminum was introduced with stirring into a melt of TNT, ammonium nitrate and water. After stirring for 30 minutes, the mix was poured into the bulb of the manometric testing apparatus.

In most cases, the initial evolution of gas was faster than the rate finally achieved. The rates recorded here are the "steady-state" rates obtained after the first 30 minutes had elapsed.

In the actual filling of a mine, the mix will be near 80-81°C. (the freezing point of TNT); because this temperature is 20° below that at which our tests have been made, the rate of gas generation in practice will be much less than the figures given here. A large mine may, however, take as long as 24 hours to cool. Under these conditions it can be computed that using commercial materials containing small amounts of water the increase in volume due to gassing might be as much as 10 to 20%. Spewing under these conditions might well occur. However, the best results here obtained indicate that the gas evolution can probably be reduced, by one of several different methods, so that it will not cause a volume increase of more than 2%. This result is probably satisfactory. It is hoped that a method which can be used industrially can shortly be advanced. Parenthetically, it may be mentioned that the rate of gas evolution from Amatol or an aluminum-TNT mix is less than 1/5 that from the very best samples of Minol so far prepared.

An attempt to desensitize Torpex by coating the aluminum powder with synthetic rubber has not so far been successful. This work is being continued.

Preparations and Testing

Stability Tests

A total of 304 chemical stability tests was carried out. This included 141 thermal stability tests, 61 vacuum stability tests, 8 Bergmann-Junk determinations, 41 stabilizer determinations and 53 miscellaneous tests.

Miscellaneous

A sample of nitrosoguanidine has been prepared and subjected to stability and sensitivity tests. The freezing point, solvent action and other properties of Methylite 20 (80-20 nitroglycerine-dimethylphthalate) and of a duPont liquid explosive (EL 389)* have been determined.

References

1. Interim Report PT-19, page 29 ff.

2. Koski, RD4 (Hercules Report) December 20, 1943

3. White, Modern Calorimetry, Chemical Catalogue Company

4. WA-1628-14

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* See "Development of Liquid Explosives," by W. E. Lawson in this and previous PT reports.

Table I

Al.	Treatment of Al.	NH_4NO_3	Water	Remarks	% Increase in vol./hr.
Commercial	None	None	None		0.00
None	None	C.P.dried	None		0.17
None	None	C.P.dried	0.5%		0.30
Virgin	None	C.P.dried	None		1.6
Virgin	None	Com.dried	None		2.8
Commercial	None	C.P.dried		Mix wet with 0.25% water. Then dried with benzene	1.4
Commercial	None	C.P.dried		As above dried with benzene-alcohol	1.0
Commercial	None	C.P.dried		As above, dried with CCl_4	1.9
Virgin	None	Com.dried	0.5%		4.4
Commercial	None	Com.dried	None		6.7,4.6
Commercial	None	Com.dried	0.25%		5.4
Commercial	None	C.P.dried	0.25%		1.4
Commercial	None	Commercial	0.5%		6.0
Commercial	None	C.P.	0.5%		2.7
Virgin	None	Commercial	0.5%		5.5,6.0
Virgin	None	Commercial	0.5%		5.8
Virgin	None	Commercial	0.5%		4.7
Virgin	None	Commercial	0.5%		7.7
Virgin	None	Commercial	0.5%		8.0
Commercial	None	Commercial	0.5%	1% $Mg(NO_3)_2$ added	9.5
Virgin	None	Commercial	0.5%	4% $Mg(NO_3)_2$ added	2.9
Commercial	Heated at 400° for 4 hrs.	Commercial	None		2.1
Commercial	As above	Commercial	None	4% $Mg(NO_3)_2$ and 0.3% lecithin extract.	1.8
Commercial	None	Commercial	0.5%		7.0
Commercial	None	Commercial	0.5%	4% $Mg(NO_3)_2$ and 0.3% stearoxy acetic acid	1.0,0.8
Commercial	H_3PO_4	Commercial	0.25%		0.9
Commercial	H_3PO_4-$NaHPO_4$	Commercial	0.25%		1.5
Commercial	NaH_2PO_4	Commercial	0.25%		3.5
Commercial	NaH_2PO_4-Na_2HPO_4	Commercial	0.25%		3.7
Commercial	Na_2HPO_4	Commercial	0.25%		2.7
Commercial	Na_2HPO_4-Na_3PO_4	Commercial	0.25%		1.4
Commercial	Na_3PO_4	Commercial	0.25%		0.8
Commercial	Water	Commercial	0.25%		2.2
Commercial	Na_2SiO_3	Commercial	0.25%		2.7

DEVELOPMENT OF LIQUID EXPLOSIVES

Report Submitted by
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Thermal Stability Tests

The 150°F. heat stability tests on liquid explosives started in February were continued and similar tests were started with KRL's 80-20 NG-dimethyl phthalate for comparative purposes. The results of these tests are given in Table I.

A 50-gram sample of the KL 50-35-15 NG-DNT-TNT (Centralite 0.5% of NG) composition made with polymer (#3) oil evolved 6.6 cc. of gas (150°F. and atm. pressure) in 37 days. Essentially, the same result was obtained with a sample containing rubber strips, showing that rubber has no apparent effect on the thermal stability of this explosive. From results with Pentolite (1.7 cc. of gas in 11 days) reported in Division 8 Interim Report M29-8 under this contract, it may be concluded that the 50-35-15 mixture has about the same thermal stability as Pentolite.

The 50-35-15 composition made with glycol (#19) oil had evolved less than half as much gas as the polymer oil formula after 28 days. Addition of rubber strips to the glycol oil formula increased the evolution of gas but even so, the total evolved in 28 days was somewhat less than for the corresponding polymer oil mixture. As there appears to be no tendency for accelerated gas evolution from any of these samples, the tests are being discontinued.

The stability tests on the KRL 80-20 NG-dimethyl phthalate mixture have been under way only 9 days. At the end of this period the plain sample had evolved no gas, but the one containing rubber had evolved about the same amount (less than 1 cc.) as the corresponding samples of the KL explosive.

Samples for Navy Tests*

A 500-lb. sample of each of the following compositions was made up at the Laboratory for drop tests at the Navy Explosives Investigation Laboratory at Stump Neck, Maryland:

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*Results of these tests are reported in detail in "Explosives Investigation Laboratory Report on E.I.M. No. 36", dated 4-12-44, and issued by the Explosives Investigation Laboratory, Naval Powder Factory, Stump Neck, Maryland.

ERL Compositions

80-20 #3 NG (polymer)- dimethyl phthalate (0.5% Centralite 1 in NG)
75-25 " " " " "

EL Composition

EL-389A 55-31.5-13.5 #3 NG-DNT-TNT (0.5% Centralite 1 in NG)

The EL 55% NG composition (EL-389A) was chosen in preference to a 50% composition on the basis of the cap sensitiveness and propagation tests described in PT-19 under this heading.

Thirty 5-gallon Army Air Corps high octane gasoline cans were used as containers for the samples.

Approximately 50 lb. of liquid explosive was placed in each 5-gallon can, leaving 1 to 1-1/2 in. freeboard. The steel screw cap was replaced by a No.13 rubber stopper, which was carefully secured with wire binding, and each can was surrounded by about one inch of pulp in a wooden box for shipment to Stump Neck by Navy truck.

Tests in which the cans were dropped from a height of 47 ft. onto one-inch armor plate were started at Stump Neck March 30, and were completed on April 7. All three explosives gave one detonation at 47 ft. and one at 40 ft. The EL explosive, EL-389A, also gave one at 30 ft. The results are summarized in Table II.

The results of these tests were very surprising in that no difference was shown between the two ERL mixtures, and EL-389A, which was the least sensitive as judged by blasting cap tests (see PT-19), appeared to be slightly more sensitive to shock than the ERL mixtures.* It is possible that variations in strength of the cans had a greater effect on the results than differences between the explosives.

- -

*The possibility of detonations occurring because of explosive remaining on the armor plate from spillage during earlier drops is excluded by the following quotation from the Explosives Investigation Laboratory Report E.I.M. No.36, 4-12-44, page 3:
"6. The possibility that the detonations in these tests may have been caused by residue liquid explosive from former partial detonations or ruptures of containers, either on or under the steel plate on which the containers were dropped, is precluded by these considerations: (a) After each detonation it was necessary to replace the steel plate with a new one. (b) Each detonation resulted in a crater which was refilled with fresh earth before the next drop. (c) In the case of ruptured containers, all liquid explosive was disposed of by burning, and the steel plate and earth under it was replaced as in the case of detonations".

Future Program

An attempt will be made to determine the relative impact sensitiveness of the two ERL mixtures and EL-389A. Additional samples of these explosives may be supplied for further tests by the Navy, but their program is not known at this time.

Table I

Mark 8 Stability Tests at 160°F.

(Determined by Mercury Displacement Test -
cc. gas at 160 F. and atm. pressure/50-gm. sample)

Time (Days)	50-35-15 NG-DNT-TNT* Polymer Oil (#3) Normal	50-35-15 NG-DNT-TNT* Polymer Oil (#3) With Rubber Strips	50-35-15 NG-DNT-TNT* Glycol Oil (#19) Normal	50-35-15 NG-DNT-TNT* Glycol Oil (#19) With Rubber Strips	ERL 80-20* #3 NG-Dimethyl Phthalate Normal	ERL 80-20* #3 NG-Dimethyl Phthalate With Rubber Strips
1	0	0	0	0	0	0
6	0	0	0	0	0	0.088
9	1.07	.95	0	.36	0	.74
14	1.94	1.90	.45	1.08		
16	2.45	2.47	.66	1.39		
18	2.84	2.84	.83	1.60		
20	3.40	3.32	1.10	1.96		
25	4.05	3.97	1.54	2.43		
28	4.71	4.64	1.93	2.83		
30	5.24	5.20				
34	6.05	6.13				
37	6.58	6.72				

* All explosives contained 0.5% Centralite based on NG content.

Table II

Drop Tests at Stump Neck
5-Gallon Cans of Mark 8 Explosives

Exp. 369A	Can No.	Height Ft.	Position of Can	Remarks
	1	47	Vertical	Bending
	2	"	"	"
	3	"	"	Cracked near base of container.
	4	"	"	Bending
	5	"	"	Rupture of circumferential seam at base.
	6	"	"	Detonation
	7	40	"	"
	8	30	"	"
	9	20	"	Bending
	10	"	"	"
Exp. * M-75-25	1	47	Vertical	Bending
	2	"	"	Detonation
	3	40	"	"
	4	30	"	Bending
	5	"	"	"
	6	"	"	"
	7	"	"	"
	8	"	"	2" rupture of side seam near base.
	9	"	"	Bending
	10	"	"	"
Exp. * M-80-20	1	47	Vertical	Bending
	2	"	Horizontal	"
	3	"	Vertical	"
	4	"	Horizontal	Loss of stopper
	5	"	Vertical	Bending
	6	"	"	Detonation
	7	40	"	Detonation
	8	30	"	Bending
	9	"	"	"
	10	"	"	"

* ERL Compositions

STUDIES OF THERMAL STABILITY TESTS

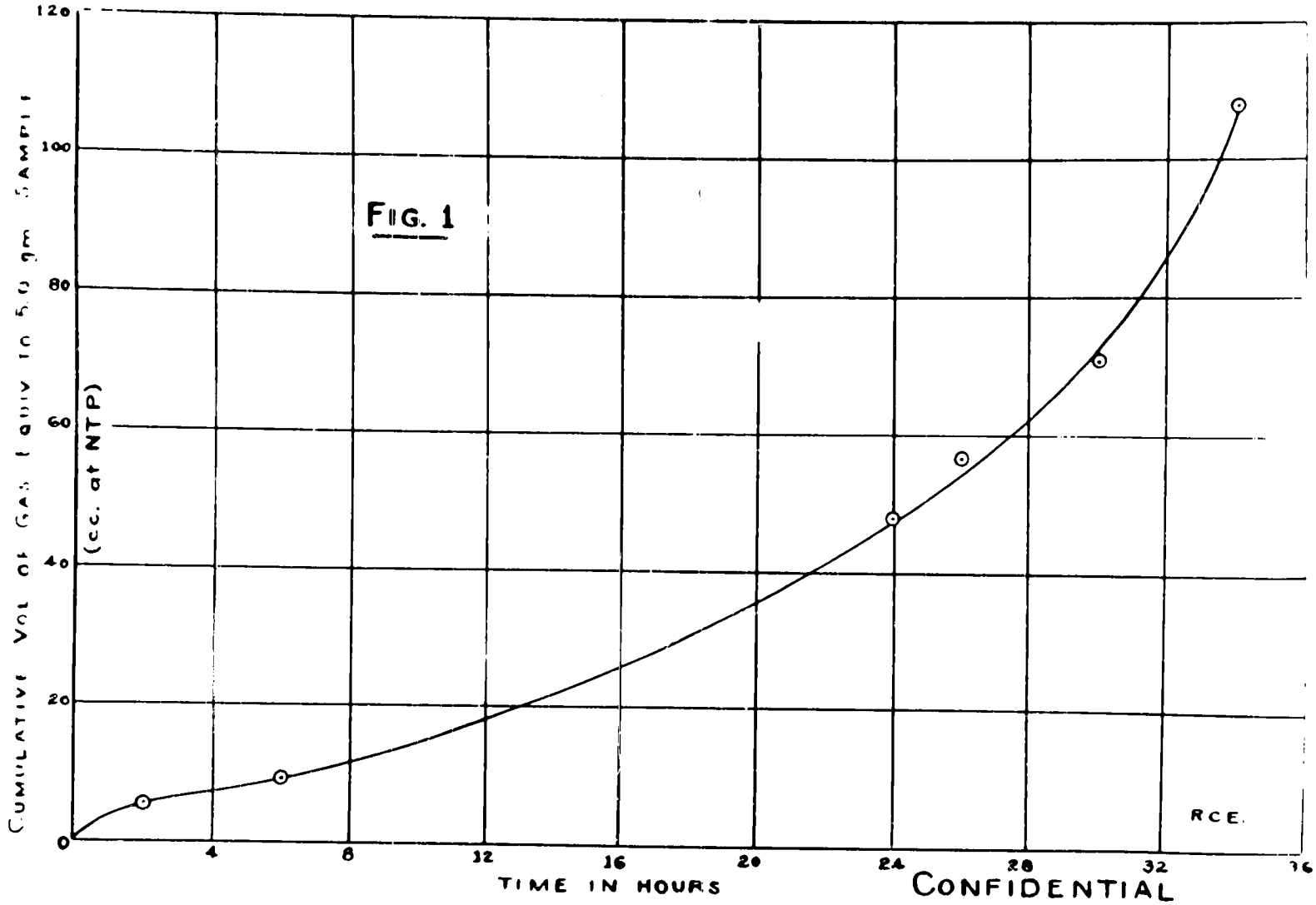
Report Submitted by
R. C. Elderfield
Columbia University

Most of the month has been devoted to checking and rechecking data previously reported on PETN and Pentolite. This is now complete and will be incorporated in a formal report to be submitted within two weeks.

The following new data on Haleite (Picatinny, November, 1943) has been obtained.

Temperature of test: 140°C. Wt. of sample, 0.5 g., dried 24 hrs. at 100°C. Gas evolution data calculated equivalent to a 5.0 g. sample and plotted on Figure I. Analysis of evolved gas as follows:

	After 24 hrs.	After 34 hrs.
Oxygen	0.0%	0.0%
Moisture (removed either by KOH or P_2O_5)	7.9	5.3
Basics	0.0	0.0
Total Acidics	2.3	6.1
NO	2.3	3.1
CO_2 and NO_2	0.0	0.0
CO	9.5	8.9
Other combustibles	1.5	1.0
N_2O	60.9	34.2
Nitrogen	17.9	44.5



POLAROGRAPHIC INVESTIGATION OF EXPLOSIVES

Report Submitted by
J. J. Lingane
Harvard University

Diffusion Current Constants of Mercury and Lead

Mercuric fulminate and lead azide are common components of primers and primer-detonators, along with antimonous sulfide, potassium chlorate, abrasive, etc., and both of these compounds are soluble in ammonium acetate solutions. Hence in the method of analysis that we are developing it is planned to extract mercuric fulminate and lead azide (together with potassium chlorate, barium nitrate, etc.) from the primer sample by leaching with a rather concentrated ammonium acetate solution, and thus effect a separation from antimony sulfide and other insoluble substances. For the polarographic determination of the mercury and lead the ammonium acetate extract is acidified with hydrochloric acid, diluted to a known volume, and the concentrations of mercury and lead are evaluated simultaneously from a polarogram of this solution.

In a supporting electrolyte composed of 1N hydrochloric acid plus 1N ammonium chloride plus 1N acetic acid plus 0.01% gelatin, which corresponds to the solutions obtained by acidifying the ammonium acetate extract with hydrochloric acid, the half-wave potential of lead is -0.45 v. vs. the S.C.E. and the diffusion current is excellently developed. The mercury wave in the same supporting electrolyte starts right from zero applied e.m.f., and a very well defined diffusion current is obtained well in advance of the lead wave. The diffusion currents of both metals are directly proportional to their concentrations when proper correction is made for the residual current (see Tables I and II).

To avoid the necessity of individual calibrations for each analysis we have determined the diffusion current constants,

$$i_d/Cm^{2/3}t^{1/6},$$

of both mercuric mercury and lead in supporting electrolytes that match the solution that will be obtained in an actual analysis. The data obtained are shown in Tables I and II. With these values available the concentrations of mercury and lead can be computed directly from the observed diffusion

currents, and the known m and t values of the capillary used, by means of the equation[1]

$$C = \frac{i_d}{I\, m^{2/3} t^{1/6}} \qquad (1)$$

where I represents the diffusion current constant; i.e., the actual value of diffusion current when C = 1 millimolar and $m^{2/3}t^{1/6}$ = 1 mg.$^{2/3}$sec.$^{-1/2}$,

Table I

Diffusion Current Constant of Lead

$m^{2/3}t^{1/6}$ = 2.00 mg.$^{2/3}$sec.$^{-1/2}$ Temp. = 25°C,

The diffusion current values have been corrected for the residual current of the supporting electrolyte alone. 0.01% gelatin used as a maximum suppressor.

Pb Millimolar	i_d Microamp,	$\frac{i_d}{Cm^{2/3}t^{1/6}}$
(Series A, supporting electrolyte 1.2 M HCl, 3.1 M NH_4Cl, 3.1 M acetic acid)		
0.108	0.76	3.52
0.312	2.14	3.43
0.503	3.75	(3.73)
0.763	5.28	3.46
0.930	6.38	3.43
1.140	8.03	3.52
1.275	9.00	3.53
		Av. 3.48
(Series B, supporting electrolyte 1.0 M HCl, 1.0 M NH_4Cl, 1.0 M acetic acid)		
0.506	3.73	3.69
0.928	6.85	3.69

[1] J.J. Lingane, Ind. Eng. Chem. Anal. Ed., 15, 583 (1943),

Table II

Diffusion Current Constant of Mercury

$m^{2/3}t^{1/6} = 1.99$ mg.$^{2/3}$sec.$^{-1/2}$ Temp. = 25°C

The diffusion current values have been corrected for the residual current of the supporting electrolyte alone. 0.01% gelatin present as a maximum suppressor.

Hg^{II} Millimolar	i_d Microamp.	$\frac{i_d}{Cm^{2/3}t^{1/6}}$
(Series A, supporting electrolyte 1.2 M HCl, 3.1 M NH_4Cl, 3.1 M acetic acid)		
0.425	3.00	3.54
1.69	12.20	3.62
3.99	28.13	3.54
7.32	52.1	3.54
		Av. 3.56
(Series B, supporting electrolyte 1.0 M HCl, 1.0 M NH_4Cl, 1.0 M acetic acid)		
0.101	7.82	3.87
4.03	30.7	3.82

It is a well known fact that +2 mercury in hydrochloric acid solution tends to be reduced to mercurous chloride by metallic mercury according to $HgCl_4^{=} + Hg = Hg_2Cl_2 + 2\ Cl^-$. Both the speed and extent of reduction is, of course, smaller the larger the concentration of chloride ion in the solution, since the tetrachloro mercuric ion is stabilized by excess chloride ion. Since mercury from the dropping electrode collects in the bottom of the polarographic cell, experiments were made to determine if the above reaction takes place rapidly enough in 1 M hydrochloric acid to interfere with the polarographic determination of mercury. A 5 millimolar solution of mercuric chloride in 1 M hydrochloric acid was placed in the cell, freed from dissolved air with nitrogen, and the dropping electrode was then introduced. The diffusion current of the mercury was observed as a function of time. Under these conditions no appreciable change in the diffusion current was observed over a period of 30 minutes, although the mercury that collected in the bottom of the cell acquired a dark film (mixture of mercurous chloride and mercury). It is evident that the reduction of

small concentrations of mercuric mercury in 1 N hydrochloric acid does not take place rapidly enough to interfere with the polarographic determination of mercury, provided that the diffusion current is measured without delay after the dropping electrode is placed in the solution.

Determination of Antimony Sulfide

Antimonous sulfide will be left in the residue from the extraction of the primer sample with ammonium acetate solution. In previous reports it has been shown that antimonous sulfide can be dissolved fairly easily in a hot alkaline tartrate solution composed of 1 N potassium hydroxide and 2 N potassium tartrate. Although the cathodic wave of the antimonyl tartrate complex present in such a solution is well separated from the anodic sulfide wave it is not possible to accurately determine the antimony and sulfide directly from a polarogram of this solution because each of these substances exerts a deleterious effect on the wave of the other. (See PT-19, page 64 ff.). Therefore it is necessary to separate the antimony and sulfide if an accurate polarographic determination of each of them is to be made. For the determination of the antimony the alkaline tartrate extract may be acidified strongly with hydrochloric acid (2 to 4 N excess), boiled to redissolve precipitated antimony sulfide and expel hydrogen sulfide, and then diluted to a known volume with addition of sufficient hydrochloric acid so that its final concentration is 1 N. A polarogram of this solution shows an excellent antimony wave whose half-wave potential is -0.15 v. vs. the S.C.E.

Table III contains data for the diffusion current constant of antimony in a supporting electrolyte composed of 1 N hydrochloric acid plus 0.2 N tartaric acid plus 0.3 N potassium chloride plus 0.01% gelatin, which corresponds to the solution that would be obtained by acidifying 10 cc. of the alkaline tartrate extract with hydrochloric acid and diluting to 100 cc.

Table III

Diffusion Current Constant of Antimony

$m^{2/3}t^{1/6}$ = 2.00 $mg.^{2/3}sec.^{-1/2}$ Temp. = 25°C.

The diffusion currents have been corrected for the residual current

Sb Millimolar	i_d Microamp.	$\frac{i_d}{Cm^{2/3}t^{1/6}}$
0.773	7.19	4.64
2.47	22.6	4.58
3.07	27.6	4.52
4.40	39.6	4.50
	Av.	4.56

Experiments are in progress to determine the sulfide by collecting the hydrogen sulfide that is evolved when the alkaline tartrate extract is acidified with hydrochloric acid. An apparatus has been assembled which permits the absorption of the hydrogen sulfide in dilute sodium hydroxide solution; the sulfide ion in this solution can then be determined either polarographically or by iodometric titration. A number of preliminary experiments have been carried out using pure zinc sulfide as a source of sulfide, to test the completeness of recovery of the evolved hydrogen sulfide. So far the results have not been very encouraging. We have found that it is difficult to obtain complete absorption of hydrogen sulfide in the sodium hydroxide receiving solution. Furthermore in alkaline medium sulfide is oxidized quite rapidly by atmospheric oxygen, which necessitates protection of the solution with nitrogen or other inert gas. However, we believe that most of the difficulties have been met and they are in the process of being circumvented. Since these experiments are still in a state of flux and are being continued we shall defer a detailed report on them until later.

POLAROGRAPHIC EXAMINATION OF EXPLOSIVES

Report Submitted by
V. W. Meloche
University of Wisconsin

In examining members of the diphenylamine nitration series, it has been necessary to make certain changes in procedure as the work has progressed to the more highly nitrated compounds. In our last report (PT-19, page 67 ff.), mention was made of maxima occurring in reduction curves of 4- nitrodiphenylamine. It was also noted that the addition of eosine would eliminate such maxima. It should be added that in all cases where eosine was used as a repressant, the residual current was not ideally defined. In seeking a substitute for eosine ethyl cellulose was tried and found to be an excellent repressant for this system. The maximum in the curve was removed and the shape of the lower part of the curve was improved. In Figure 1, curve 1 represents reduction for a solution to which no repressant was added. Curve 2 illustrates the addition of 0.01% ethyl cellulose. The addition of this substance apparently changes the value of $i_d/Cm^{2/3}t^{1/6}$. It may be presumed that if ethyl cellulose had been added to systems containing the nitroso compounds mentioned in our last report, their $i_d/Cm^{2/3}t^{1/6}$ would have been changed. The deviation is slight and has little importance until further check runs are made.

In the last report we suggested the use of tetraalkyl base as a supporting electrolyte for the dioxane system. Tetraethylammonium hydroxide was prepared and tested by means of the polarograph. In attempting to dissolve sufficient base to make the solution 0.15 molar, experiment showed that dioxane-water systems which contained 80% dioxane separated into two layers. A system containing 60% dioxane did dissolve the desired amount of tetraethylammonium hydroxide and left a reasonable working margin. Hence, the basic systems described later in this report represent 60% dioxane while the acid systems represent 80% dioxane. The results do show that different reductions take place in the basic solutions than take place in the acid solutions. If it is desirable to use a basic solution in order to identify one of these compounds, it may be desirable to standardize on a solution containing 50% dioxane and prepare all of our standard information on this basis.

Materials:

1. Dioxane ---- Purification was given in PT-19, page 68.

2. n-nitrosodiphenylamine --
   preparation given in PT-18.
3. p-nitrosodiphenylamine
   Eastman white label
4. 4-nitrodiphenylamine--
   Eastman white label was of inferior quality. Recrystallisation from an ether solution after filtration yielded a good product. m.p. 131-2°C.
5. 2-nitrodiphenylamine
   Eastman white label.
6. HCl - reagent grade
7. ethyl cellulose - courtesy of United States Forest Products Laboratory.
8. tetraethylammonium hydroxide -- Prepared by technique similar to that described for tetramethyl compound(1)

The following two tables give representative data for the reduction of 2-nitro and 4-nitrodiphenylamine.

Table I

Reduction of 4-nitrodiphenylamine

System: 80% dioxane, 20% water containing 0.01% ethyl cellulose and 0.2N HCl

Drop time at half wave: 3.90
Electrode p/m : 40.3
Pressure : 58.3
Halfwave potential : 0.40 volt vs S.E.C.

	conc. mmols/liter	i_d microamps	i_d/c	
1.	0.5	5.76	11.52	
2.	1.0	11.37	11.37	$i_d/Cm^{2/3}t^{1/6}$ = 5.97
3.	1.5	16.89	11.26	based on average
4.	2.0	22.50	11.25	
5.	2.5	27.02	11.02	

see curve 2 of Figure 1 for a typical polarogram

(1) JACS 60, 1770 (1938) Peracchio and Meloche

Table II

Reduction of 2-nitrodiphenylamine

System: 80% dioxane, 20% water containing 0.01% ethyl cellulose and 0.2M HCl

Drop time at half wave: 3.94
Electrode p/m : 40.3
Pressure : 58.3
Half wave potential : 0.39 volt vs S.E.C.

	conc. mmols/liter	i_d microamps	i_d/c	
1.	0.5	5.79	11.58	
2.	1.0	11.37	11.37	$i_d/Cm^{2/3}t^{1/6}$ = 7.00
3.	1.5	17.25	11.50	based on average
4.	2.0	22.53	11.27	value of i_d/c
5.	2.5	28.65	11.46	

The curve for the reduction of the 2-nitro compound is quite similar to that for the 4-nitro compound. (See Figure 2).

Table III

A Comparison

	$i_d/Cm^{2/3}t^{1/6}$	1/2 wave potential	
1.*	4.58	0.67	2-nitrodiphenylamine
2.*	4.62	0.67	p- "
3.	6.97	0.40	4-nitrodiphenylamine
4.	7.00	0.39	2-nitrodiphenylamine

This table represents reductions performed in acid solutions. The asterisks represent solutions to which no ethyl cellulose had been added.

Table IV may be compared to Table III, remembering that Table IV represents a system which is basic to tetraethyl ammonium hydroxide and contains only 60% dioxane.

Table IV

	$i_d/cm^{2/3}t^{1/6}$	1/2 wave potential	
1.	2.2	1.15	n-nitrosodiphenylamine
2.	2.2	1.15	p-nitrosodiphenylamine
3.	4.6	0.99*	4-nitrodiphenylamine
4.	4.1	0.90	2-nitrodiphenylamine

* a color change took place and it may be possible that an interference occurs in this reduction.

Figures III and IV represent reduction of n-nitrosodiphenylamine and 2-nitrodiphenylamine and are examples of reduction in a basic system of dioxane.

In connection with the use of dioxane, reference might be made to information on viscosity. Data taken from Kerr and Sturm indicate that when one plots viscosity vs. percent composition of dioxane in water the curve shows a maximum which is rather flat between 40 and 70% dioxane and the viscosity changes slightly between 0 and 40 and 70 to 100. This might indicate that it would be desirable to use systems for the polarographic study in the range 40 to 70% dioxane. A slight error in % dioxane would have very little effect on the magnitude of the reduction wave.

Data Pertinent to Figures

Figure 1 approx. 2 mmol/L 4-nitrodiphenylamine
1) 0.2 N HCl in 60% dioxane
2) 0.2 N HCl in 60% " plus 0.01 ethyl cellulose

Figure 2 1 mmol/L p-nitrosodiphenylamine
1 mmol/L 2-nitrodiphenylamine
*This curve shows how the two may be recorded together

Figure 3 2 mmol/L n-nitrosodiphenylamine
0.15 N tetraethylammonium hydroxide
in 60% dioxane and 0.01 ethyl cellulose

Figure 4 2 mmol/L 2-nitrodiphenylamine
0.15 N tetraethylammoniumhydroxide in
60% dioxane and 0.01 ethyl cellulose

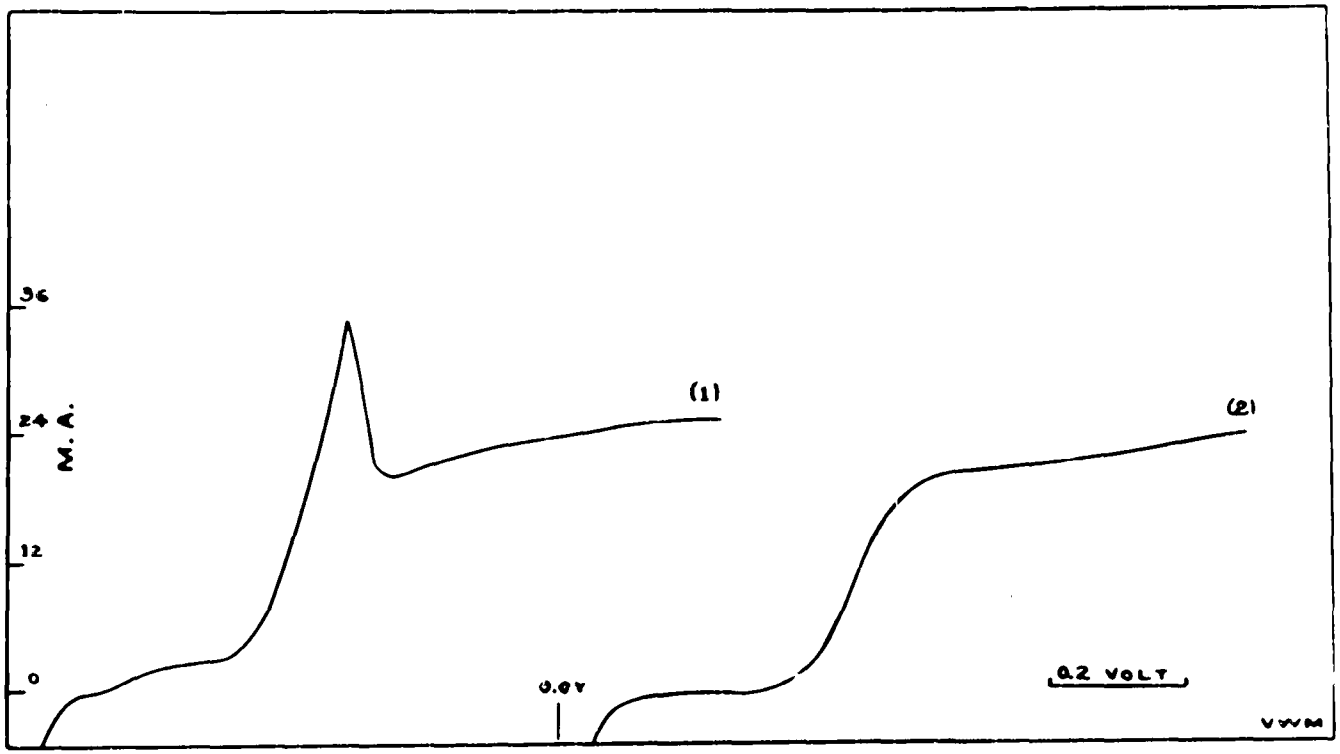


FIGURE 1

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FIGURE 2

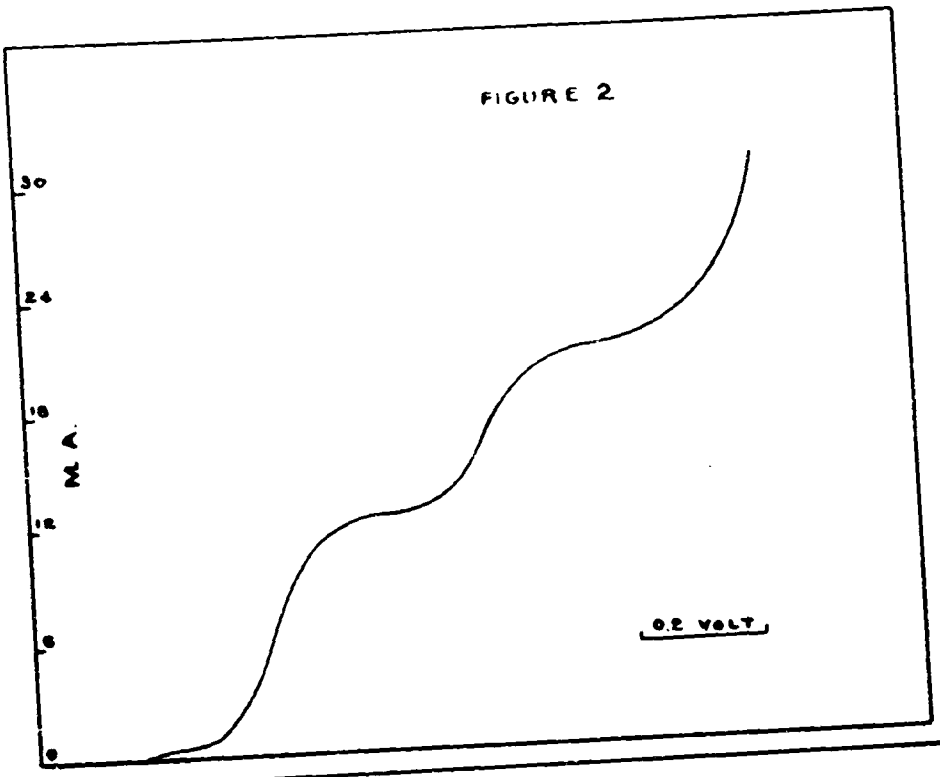
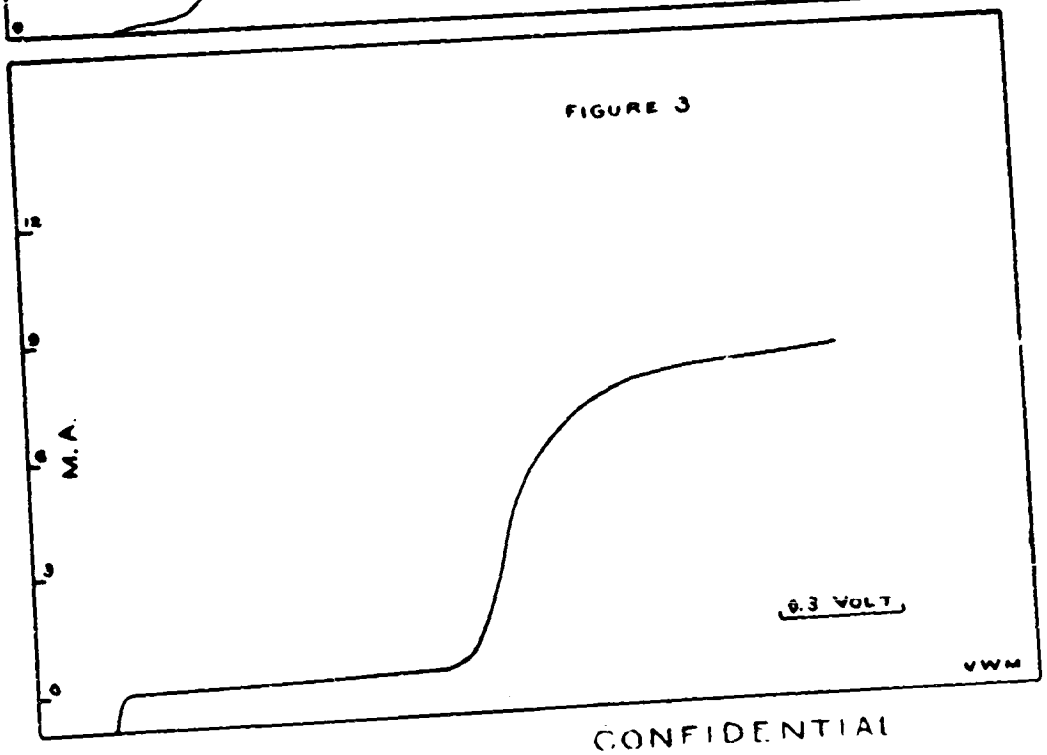


FIGURE 3



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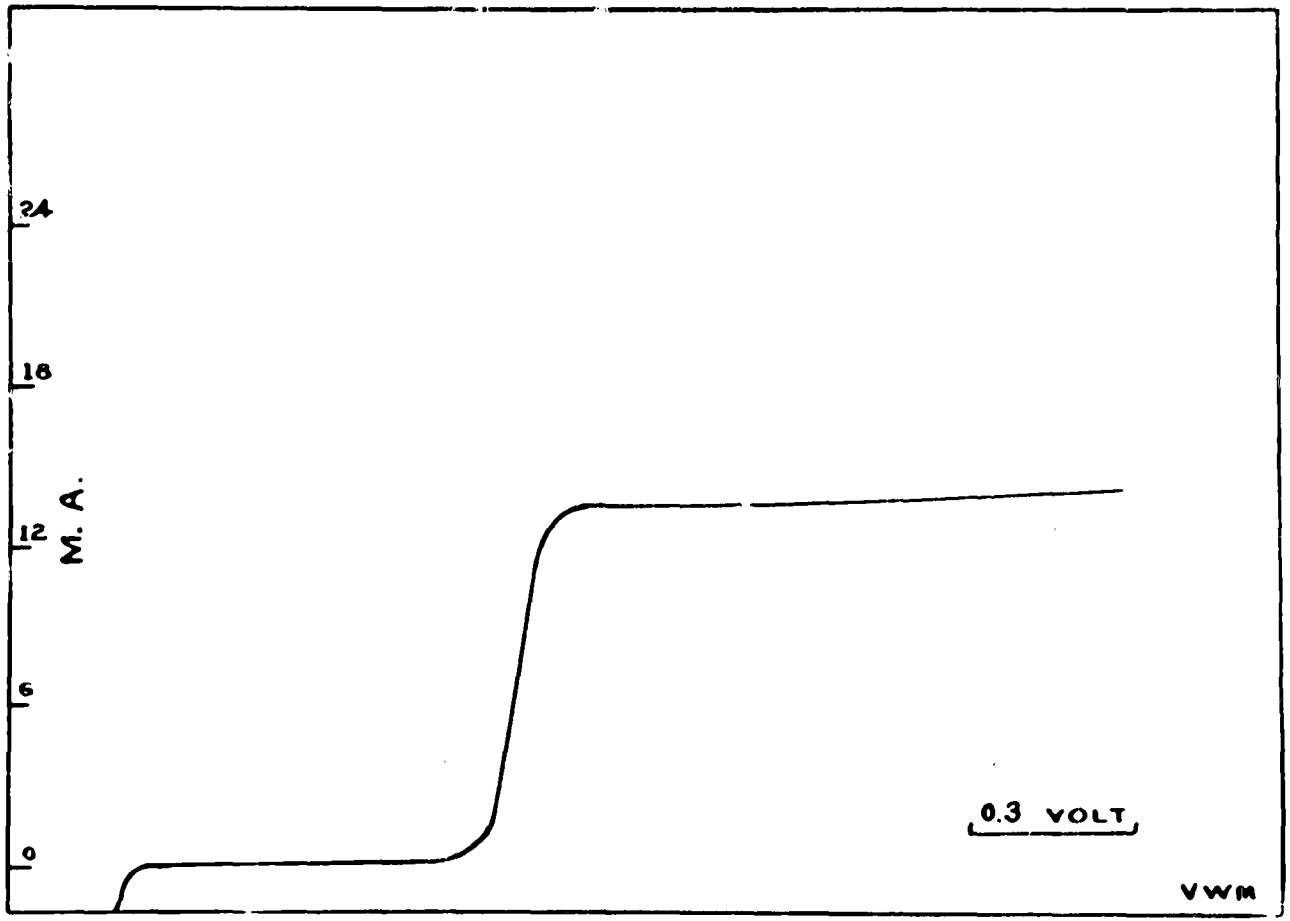

FIGURE 4

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Proposals for future work:

1. Although some further work is necessary on the nitration series of diphenylamine, it is time to request samples of aged powders. We can attempt to apply the information which we have now on such samples or we can apply the study to powders in which the aging has been accelerated by heat.

2. This study should be extended to centralites.

3. Prepare 2-nitronitrosodiphenylamine and 4-nitronitroso-diphenylamine for examination.

STI-ATI-201 327 *~~CONFIDENTIAL~~ 265650 ~~SECURITY INFORMATION~~

National Defense Research Committee, Office of Scientific Research and Development, Div. 8, Washington, D.C.

PREPARATION AND TESTING OF EXPLOSIVES, by Ralph Connor. Interim rept., 15 Mar - 15 Apr 44. 64p. illus, tables. PT-20.

Ordnance (22)	Explosives
Ammunition & Explosives (1)	Explosives - Testing

(Copies obtainable from ASTIA-DSC)

NTIS, auth: SOD Memo, 2 Aug 60 MICROFILM ~~CONFIDENTIAL~~

SoD memo, 2 Aug 60